

METALLURGIA

THE BRITISH JOURNAL OF METALS



Reduced Machining Allowance

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Vol. 4, No. 1949 - May 1950

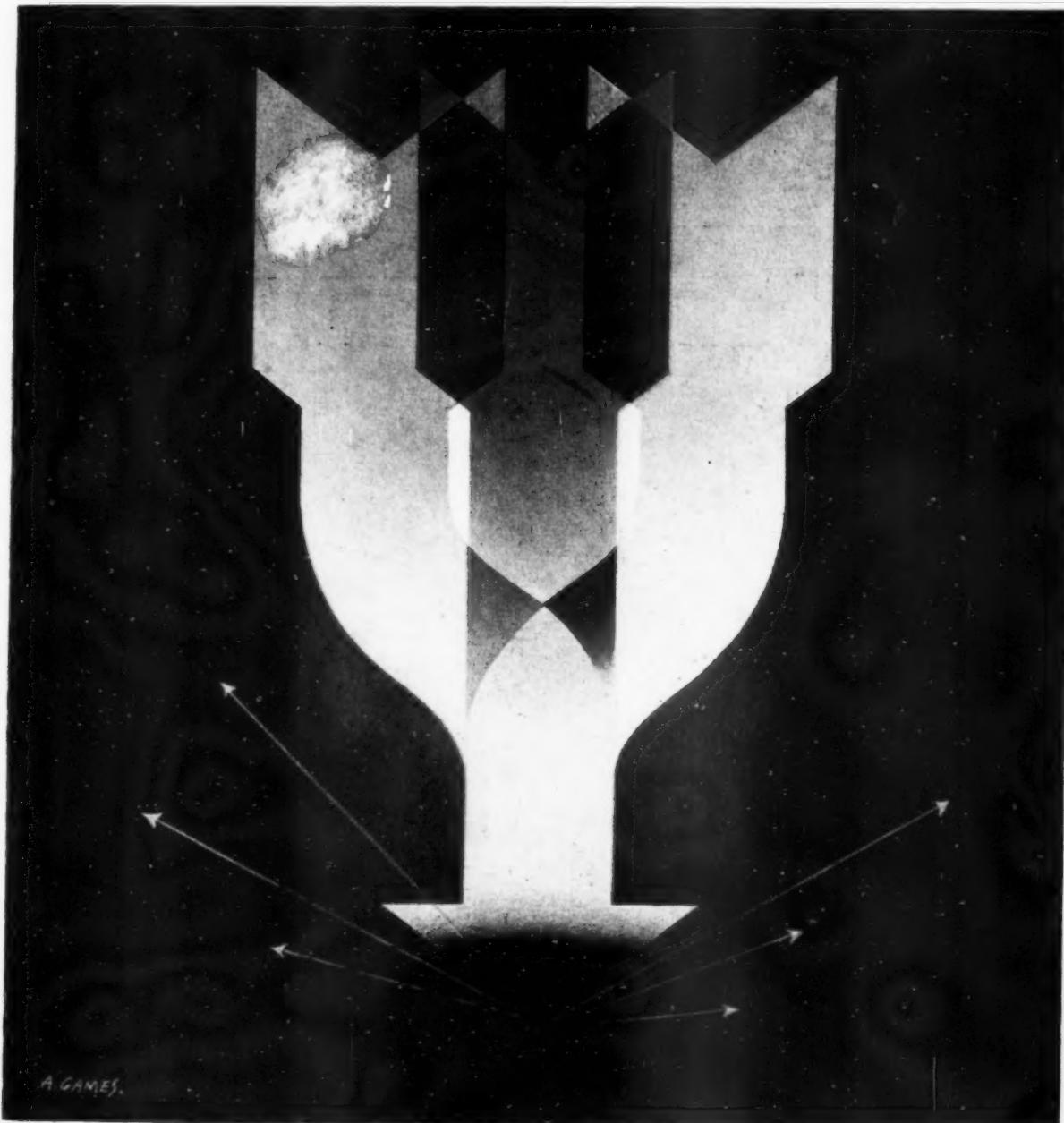
heat resisting steel castings

by

HADFIELDS
SHEFFIELD

Send for Heat Resisting Steel Brochure No. 473.

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Design by Abram Games

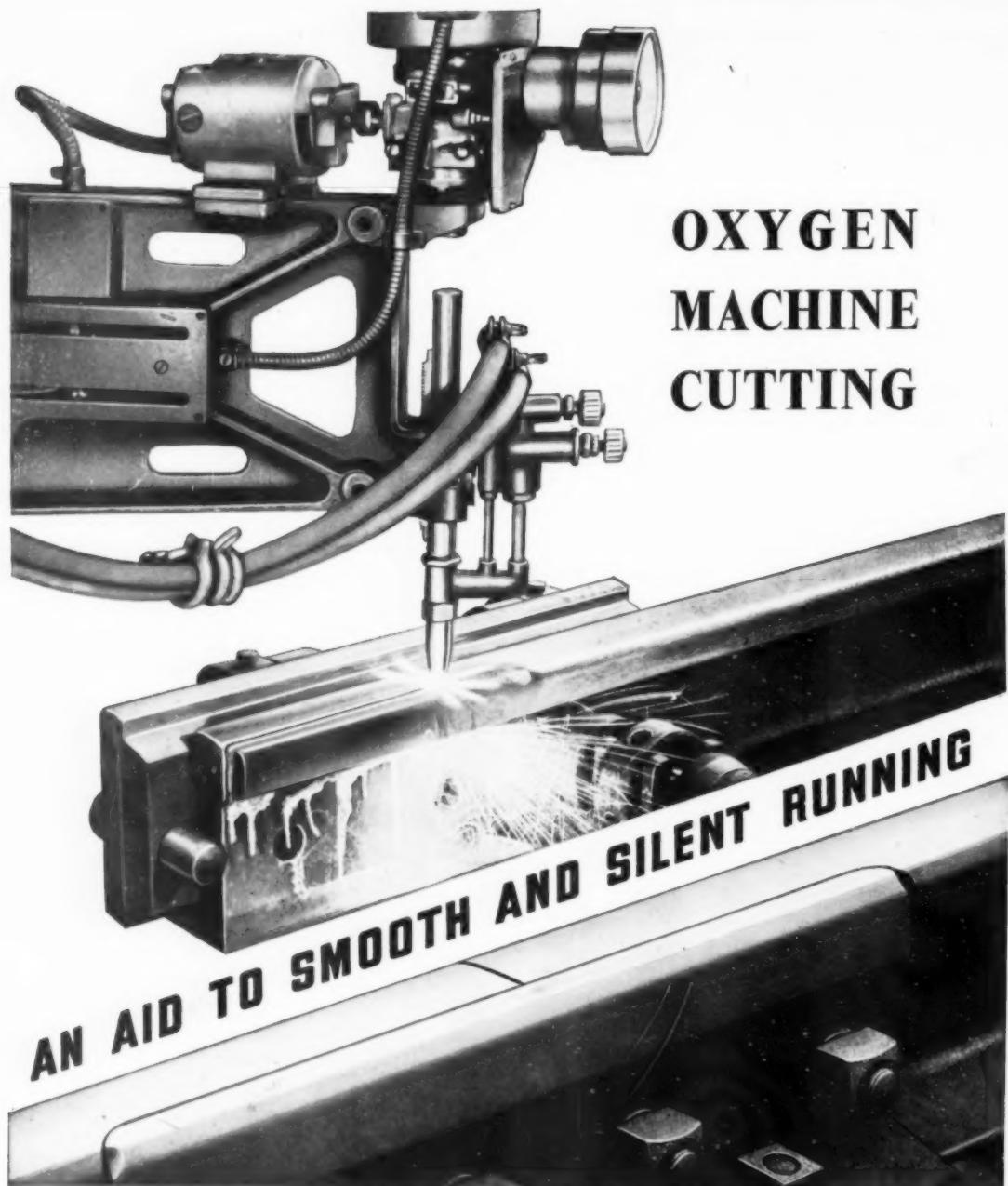
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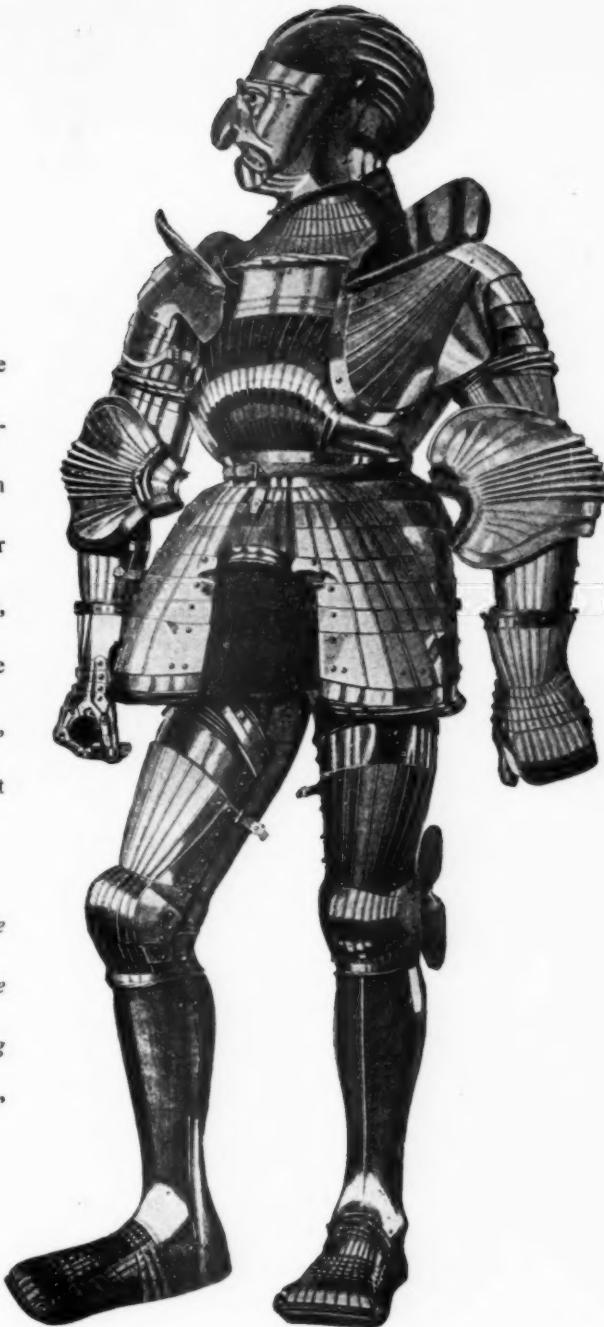
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*Armour of the Duke Christoph of Württemberg.
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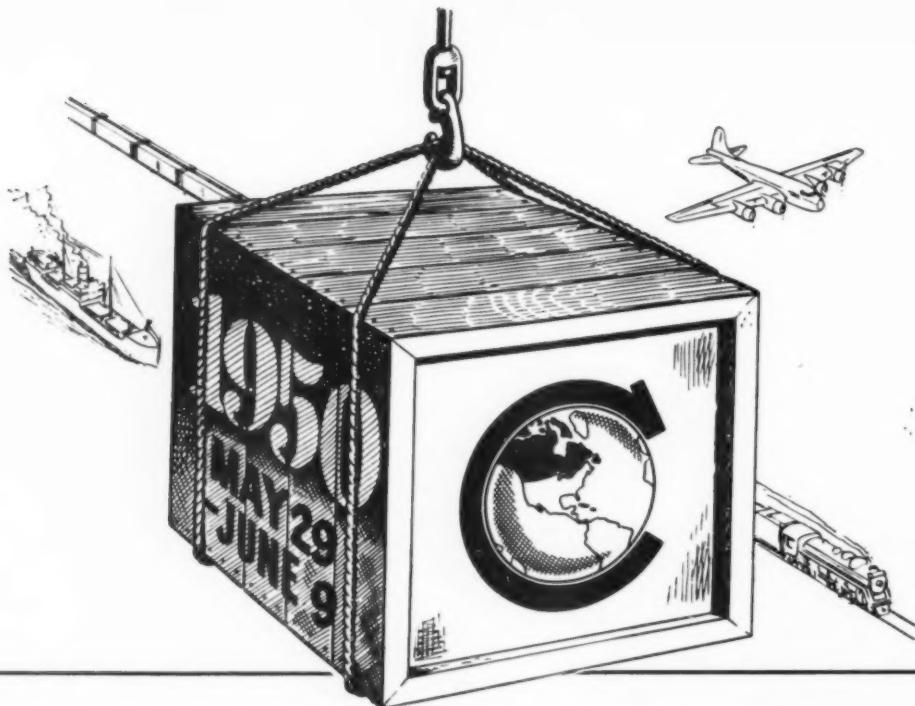
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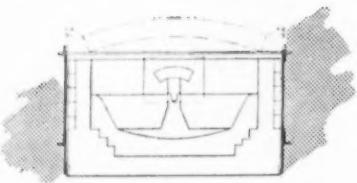
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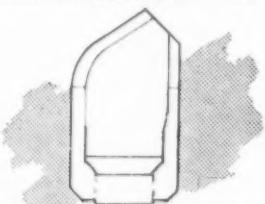
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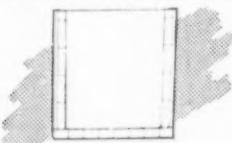
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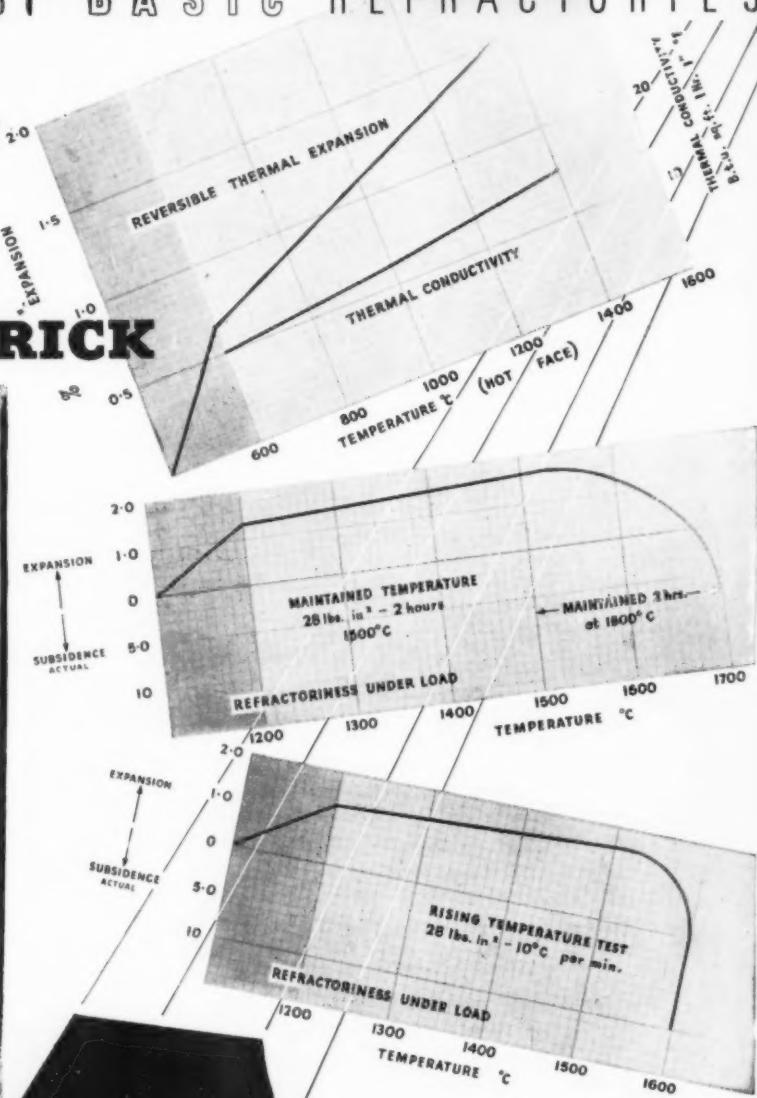
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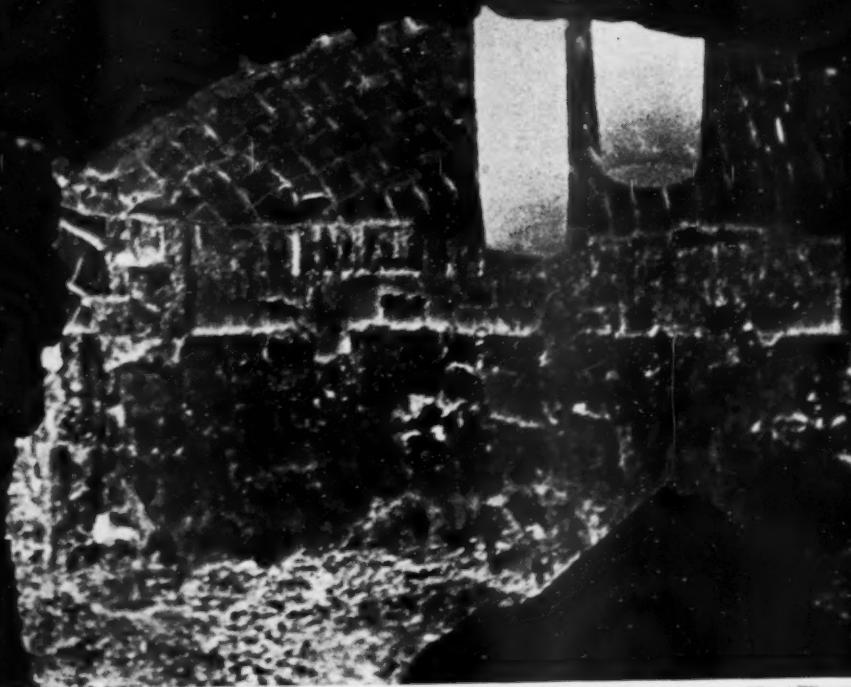


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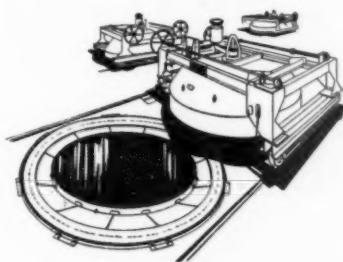


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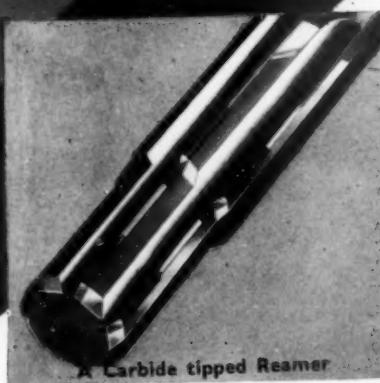
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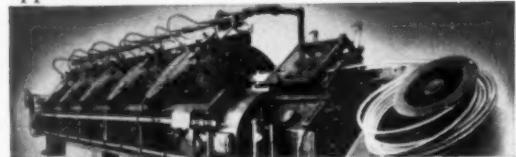
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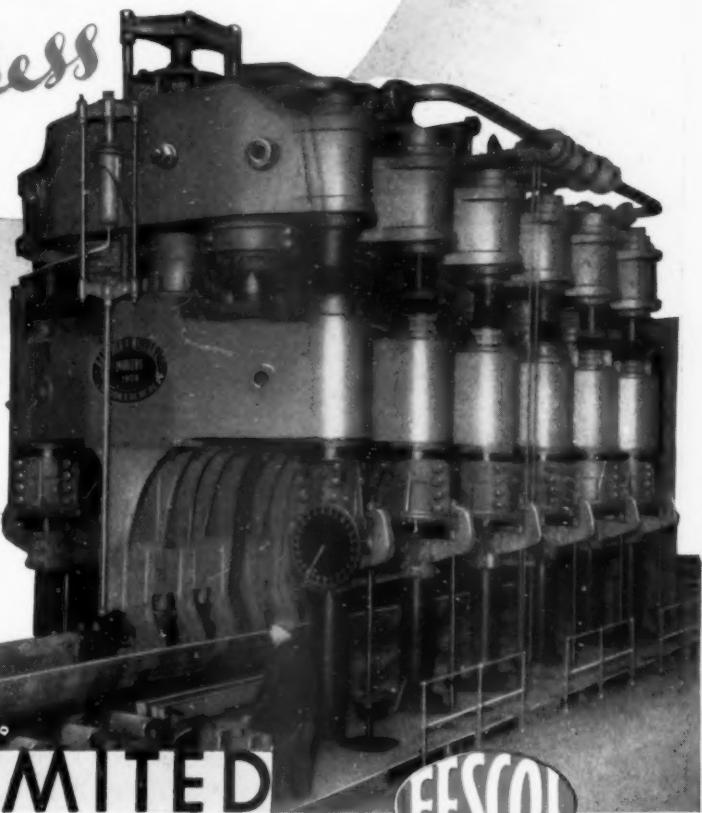
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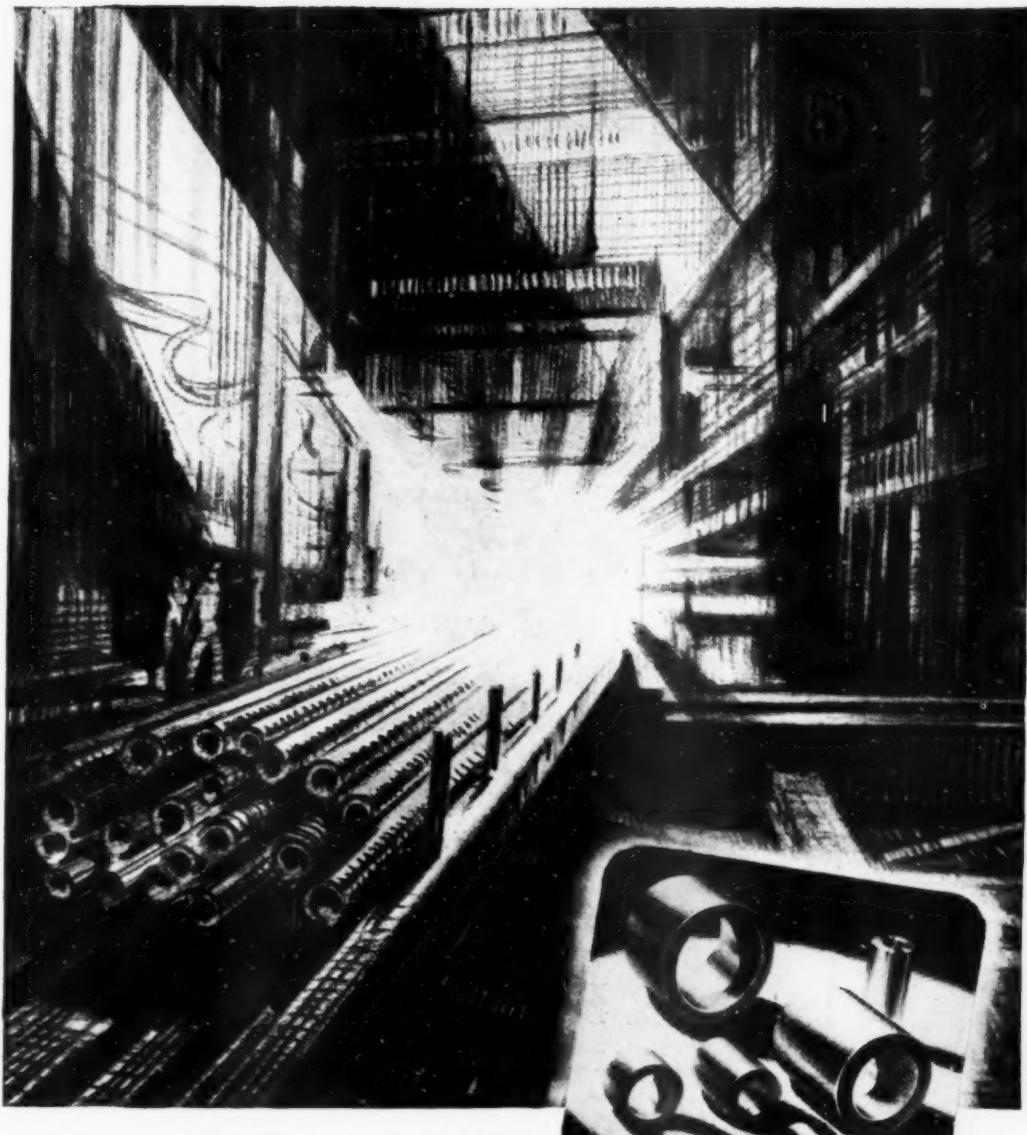
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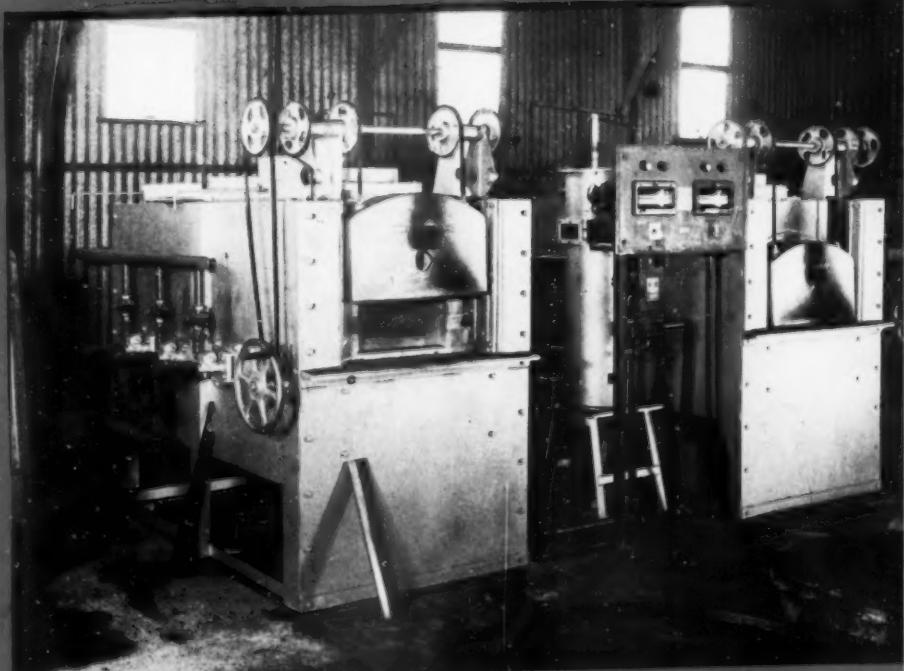
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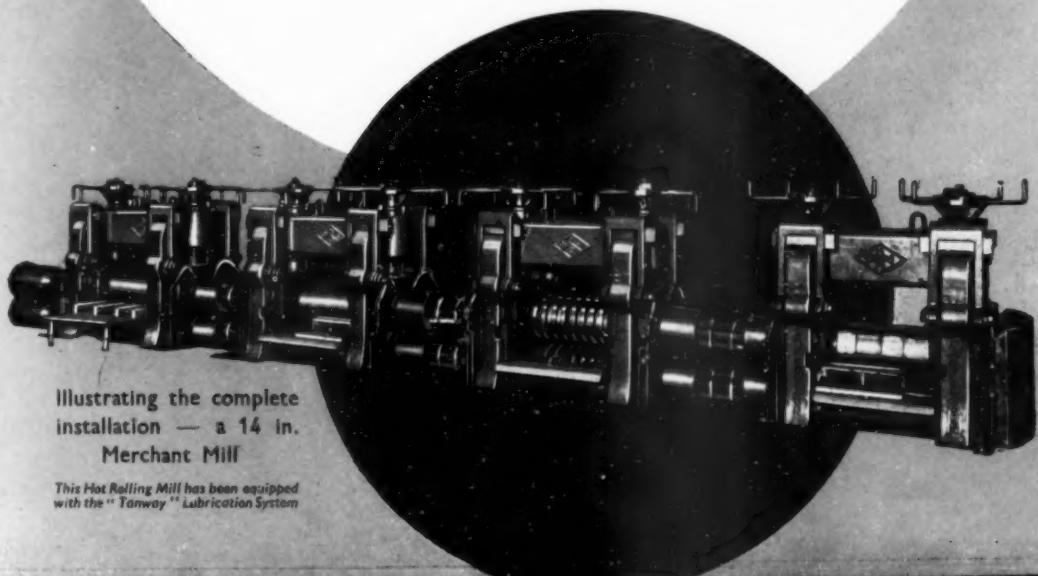
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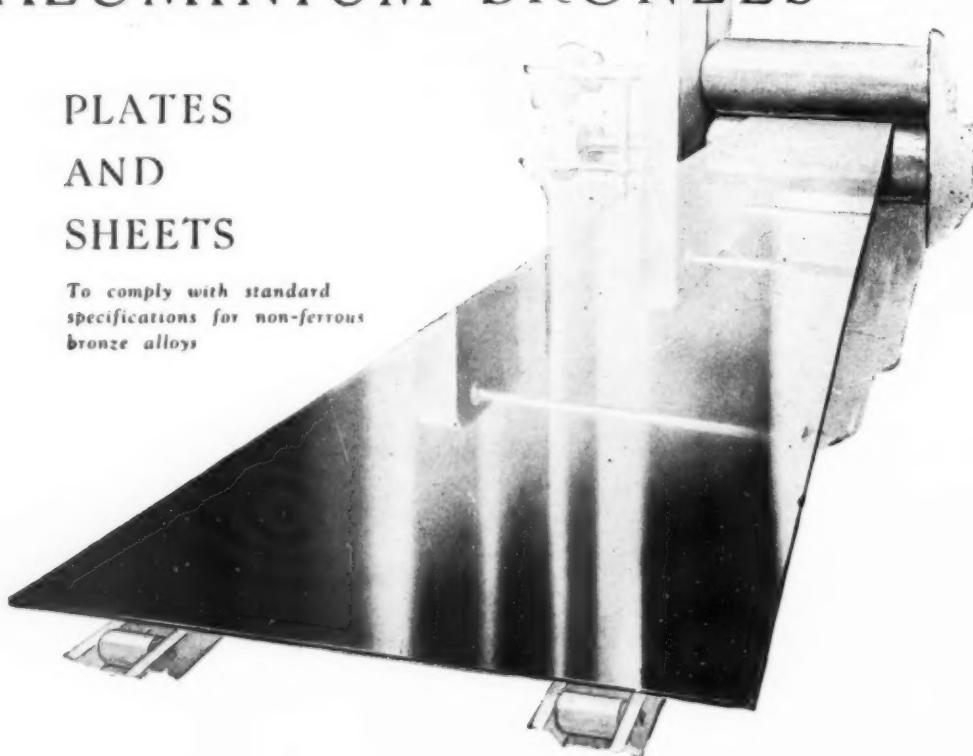
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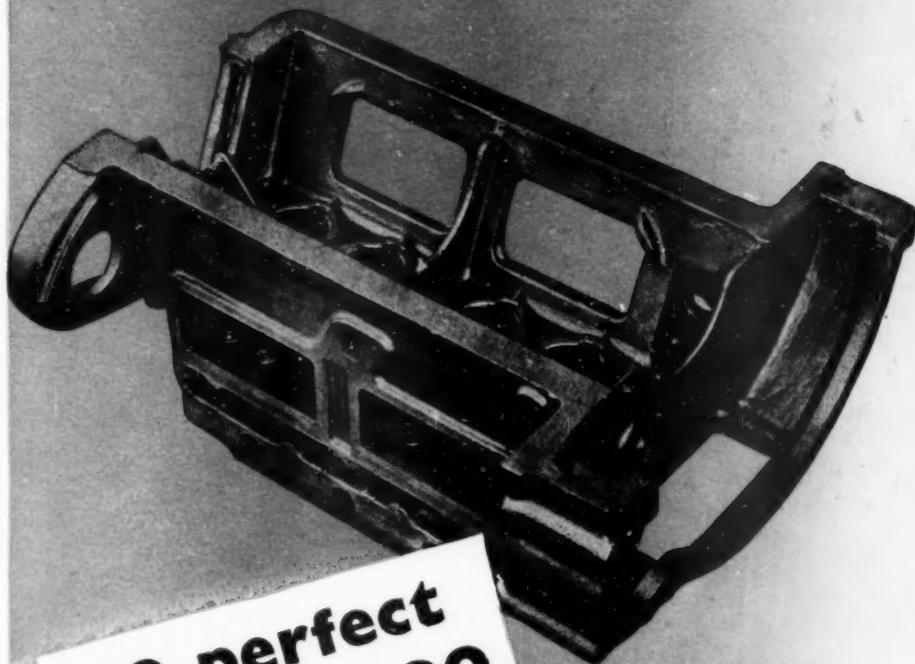
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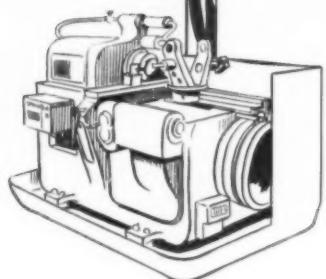
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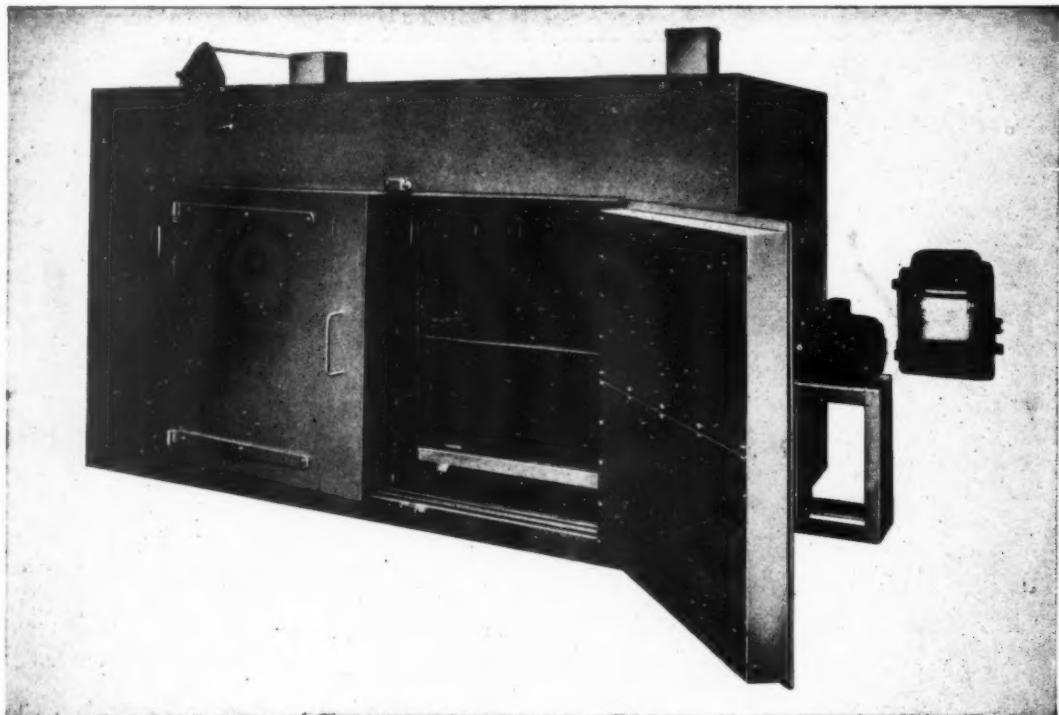
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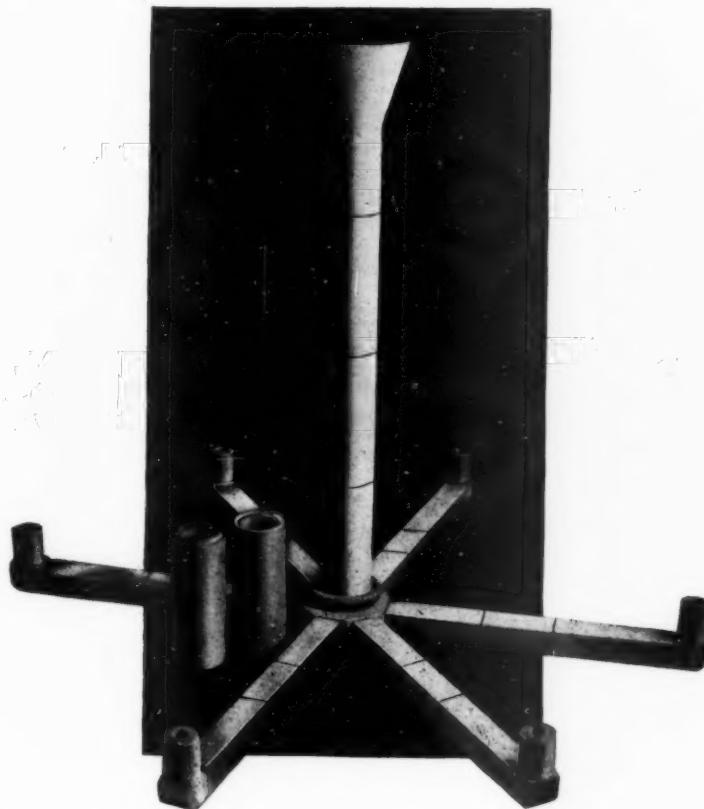
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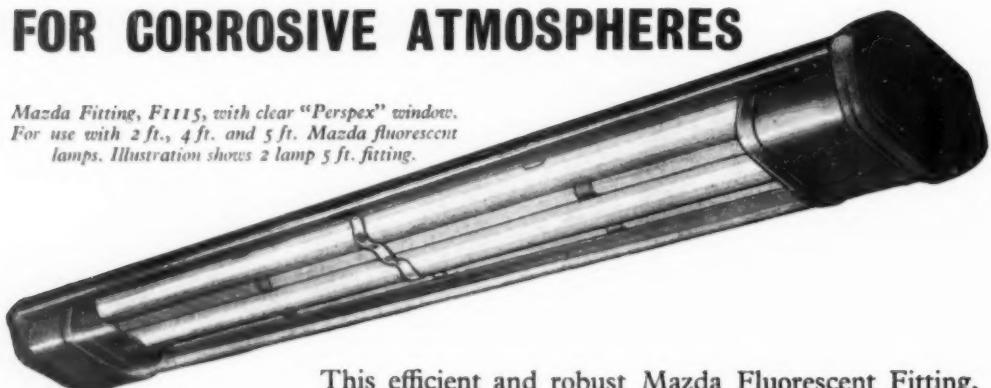
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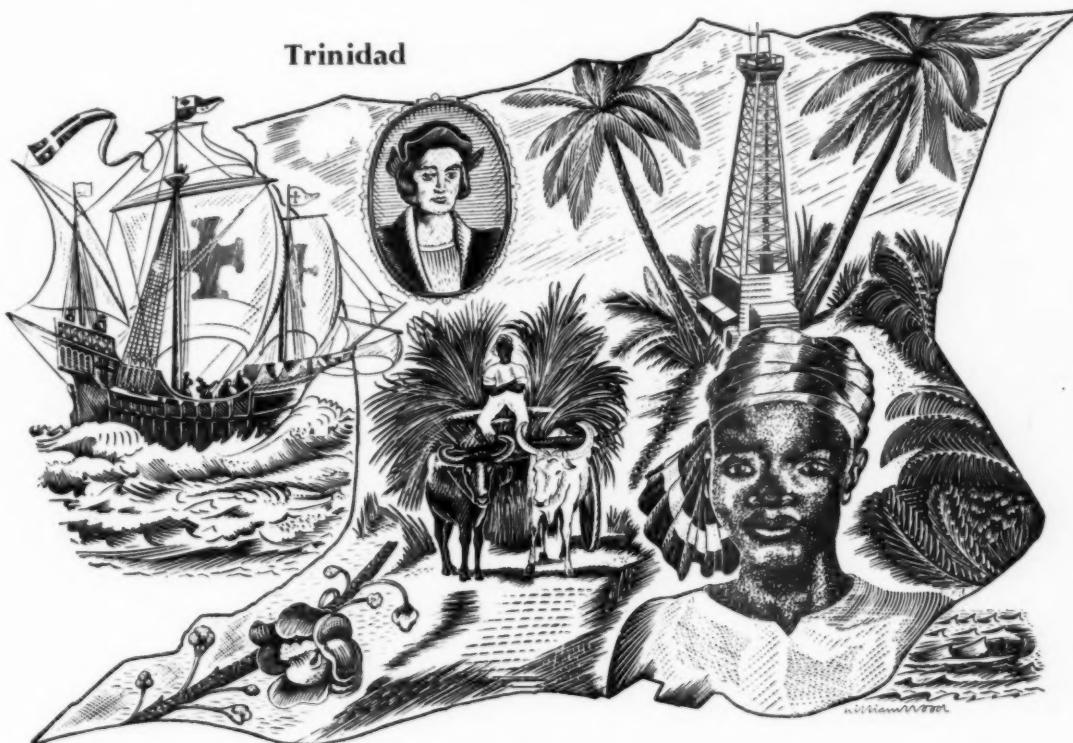
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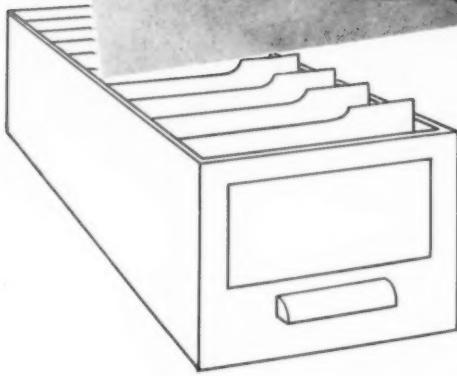
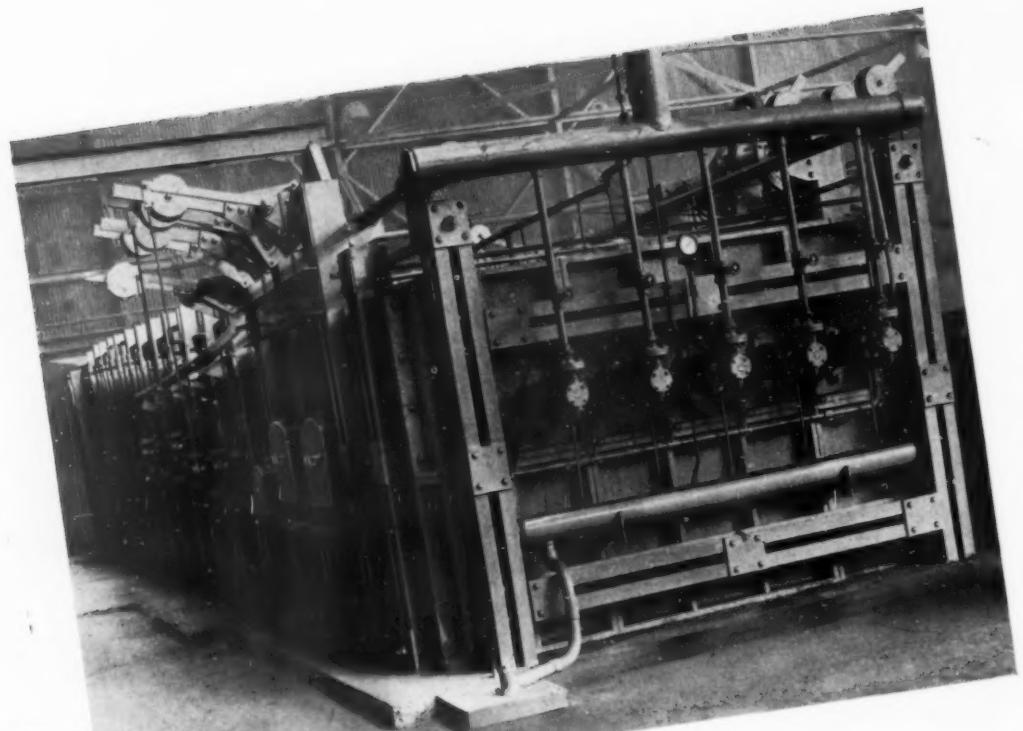
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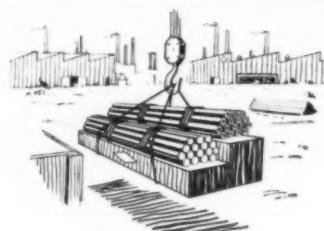
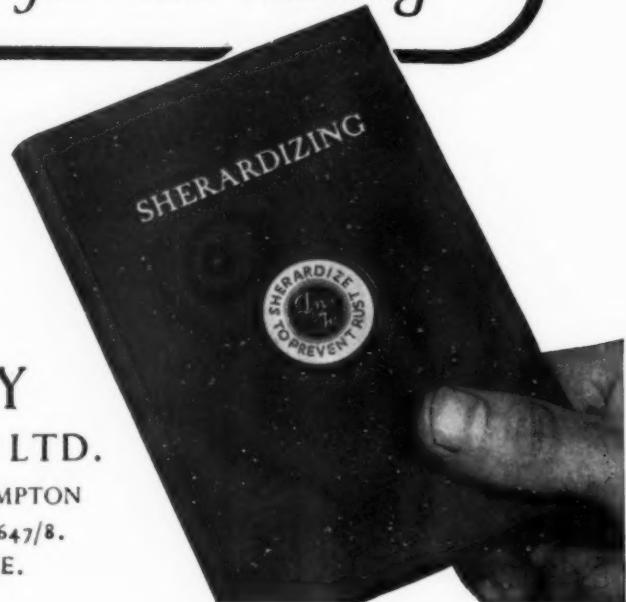
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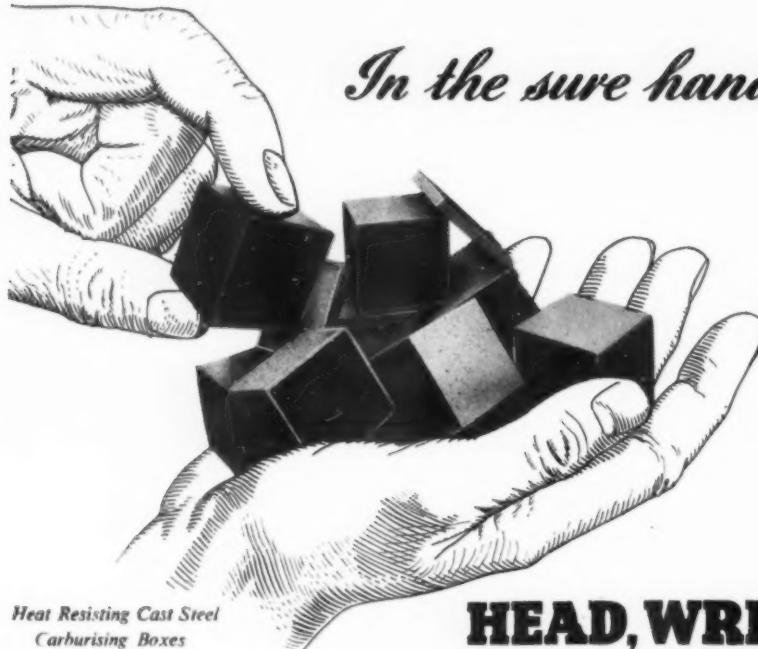
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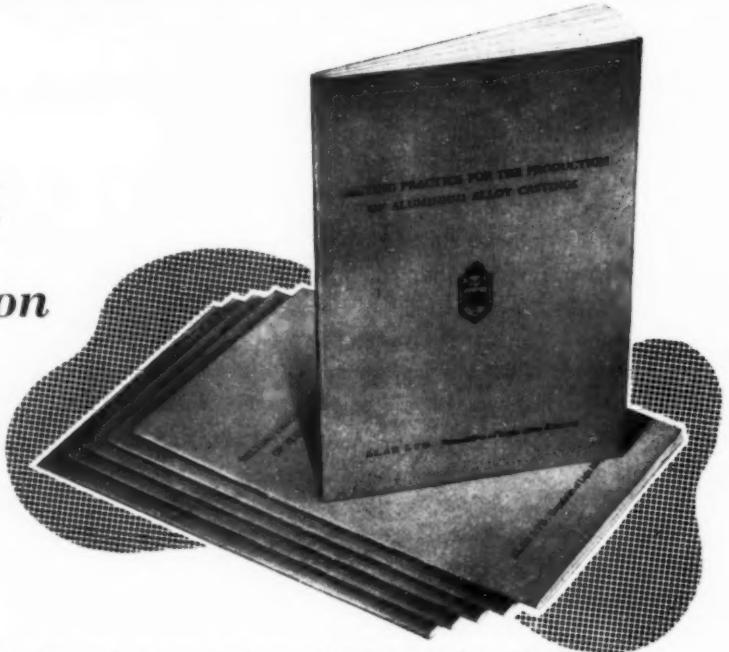
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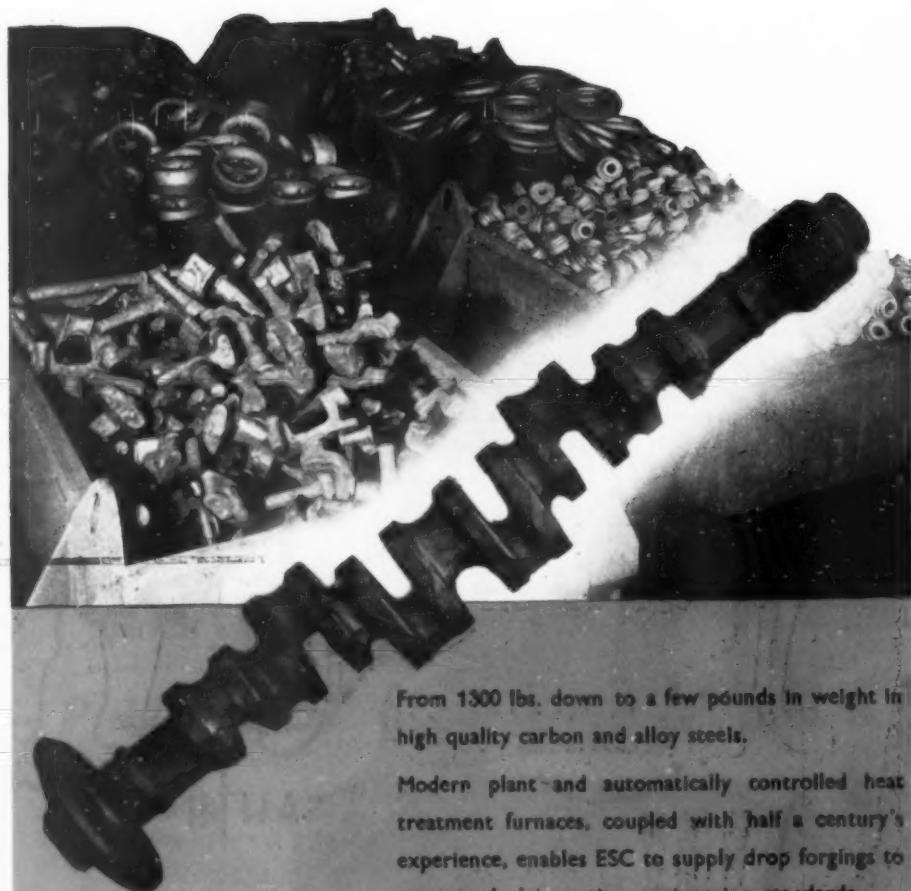
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The British Journal of Metals

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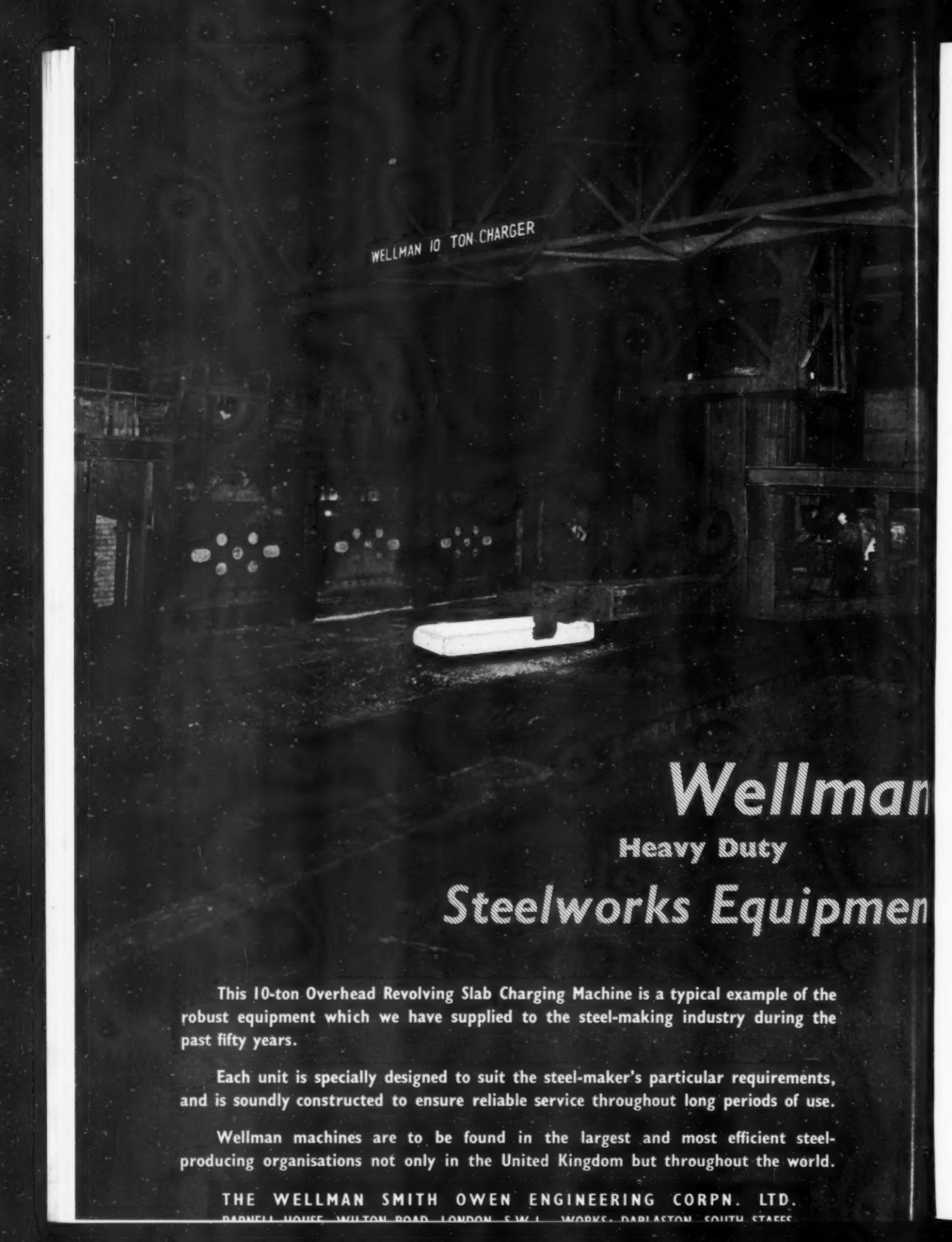
PRINCIPAL CONTENTS IN THIS ISSUE:

	Page		Page
Higher Productivity	1	Simplification in Industry	31
Original Whittle Engines Now Museum Pieces	2	Research and Production	31
Magnetic Saturation Intensities and Curie Temperatures for Some Industrial Permanent Magnet Materials. By L. Ward, M.Sc.	3-7	Cold-Worked Metals and Fatigue. By Richard Saxon	32-33
<i>An investigation is described the results of which indicate that, in alloys of the Alnico type, there is a reversible phase up to the Curie point, and the cobalt steels, as industrially heat treated, contain mix- tures of austenitic and magnetic phases.</i>		<i>Flow in cold drawing causes wear at the entrance of the die and a ring formation results; methods are given to eliminate this defect.</i>	
Corrosion and Corrosion Fatigue of Steel	7-10, 30	Recent Developments in Materials, Tools and Equipment	33-34
Capital Equipment for Metal Working <i>Recently the Loewy Engineering Co., Ltd., and F. B. Engineers, Ltd., held "open- house" at the works of the latter Company and in this article brief reference is made to some of the plant on view.</i>	11-14	<i>Roto-finishing. Magnetic Amplifiers for Thermo-Couples. Chubb Safes Rust Protected.</i>	
Some Physical Properties of Alumini- um Alloys at Elevated Tempera- tures—Part I: Thermal Conductiv- ity and Electrical Resistivity By R. W. Powell, D.Sc., Ph.D., F.Inst.P., and M. J. Hickman. Part II: Linear Thermal Expansion. By C. R. Barber, B.Sc.	15-21	Reviews of Current Literature	35-36
Italian Research on Electro-Deposition of Metals. The Work of Prof. R. Piontelli	22-25	<i>Metallic Creep and Creep Resistant Alloys. Steel Foundry Productivity. Elasticity and Anelasticity of Metals.</i>	
<i>This summary of the work carried out by Prof Piontelli covers the general electro- chemical behaviour of metals, their behaviour in concentration cells, electro- deposition of alloys, and auto-displace- ment of lead.</i>		Metallurgical Digests	37-40
Effect of Homogenising on the Struc- ture and Properties of an Ex- truded Al-Cu-Mg Alloy. By H. A. Unckel	26-29	LABORATORY METHODS SUPPLEMENT	
		This Supplement	41
		Metallurgical Education in Norway. By A. B. Winterbottom	42-44
		A Note on the Accuracy of Analysis. By E. Scheuer and F. H. Smith	44-45
		The Finch Electron Diffraction Camera	46-49
		Metallo-Organic Reagents used in Micro-Analysis—Part II. By F. Lester	49-51
		Modern Methods of Gas Analysis. By W. D. Vint	52-54
		A Split-Type Micro-combustion Furnace. By G. Ingram	54-55
		New Equipment	56-57
		Electronic Instruments Symposium and Exhibition	59-60
		Preparing Thin Specimens for Creep and Tensile Testing. By Paul Feltham, B.Sc., A.Inst.P.	60

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METALLURGIA

THE BRITISH JOURNAL OF METALS.
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NOVEMBER, 1949

Vol. XLI No. 241

Higher Productivity

HERE is evidence that our present economic position is being appreciated and efforts made to increase productivity, but a few individual cases are not sufficient to indicate a general change of outlook. Long standing inhibitions and restrictions continue to exercise a retarding influence on the expansion of the products of our industry and of our country. We are extremely conservative in our attitude to industrial practices which have been built up in our long and sometimes bitter years of industrial history, yet a profound change in this attitude is essential if we, as a nation, are to survive. What is needed is the mental freedom which enables productivity to be raised because all are attuned to the one purpose. Mr. E. T. Judge, director and chief engineer of Dorman Long and Co., Ltd., suggested that if, by the introduction of some kind of catalyst into our mental process, we were able overnight to reorient our approach to our daily work and problems to meet the country's present need, we should be fully launched on a new industrial revolution.

Mr. Judge was delivering his Presidential Address to the Cleveland Institution of Engineers. He began with a quotation from the Report of the British Steel Founders' Productivity Team, "the alternative to high productivity is starvation"—and gave his views on the subject, with special reference to the iron and steel industry. He made no apology for the steel industry's performance during recent years, since the output per worker is now about 33% above that in 1938, but approached with confidence, though critically, with a view to even better performance. Although referring more particularly to the iron and steel industry his critical analysis resulted in the expression of views that can usefully be applied to all sections of industry and we have no hesitation in passing on some of these views for careful thought.

A great urge towards producing good work in minimum time has come in the past from the knowledge that it is the way to climb the ladder to a better and a more assured job. A long period of unemployment and high unemployment benefits have largely dismissed fear as an incentive for doing a good day's work, and it must be admitted, removed some of the incentive for trying to secure appointment to a more secure job higher up the scale. Is it widely enough known, and believed, that, unless our present system of operation without the incentive of fear does lead to production at the right price and in ample quantity, we are bound to drift back to former conditions where fear, of one kind or another, produced results? We can easily arrive at a position where fear becomes the basic force in our economic system. Of course, no one wants that state of affairs, but those most vehemently opposed to it are in some

danger of bringing it about by persisting in too narrow a view of the problem.

Active enthusiasm for the job, alive from top to bottom of a department, works or industry, is an asset beyond value when high productivity is the order of the day. Fortunately it is contagious. Each enthusiast at his job has a profound effect on others in a team who are closest to him. This is equally true whether his keenness is derived from love of his work, the prospect of a fuller pay-packet, or any other cause. It is also vital that those in positions of responsibility take all possible steps to urge forward to higher productivity those whom they control, and one of these steps is an individual effort to inspire enthusiasm by work and example.

In some cases a good measure of productivity can be established by determining output per man, but the majority of us are concerned personally with the performance of small sections within an industry. Here, productivity often cannot be measured as so many tons per man per week. In such cases productivity is a question of rendering the maximum real service for the man-power expended. Of course, many factors influence the level of productivity that is obtainable, and we should still judge performance on that somewhat ambiguous term "overall efficiency." If we can establish what we call higher efficiency at all stages throughout our industry then the overall higher productivity that we seek will result.

After all, the main hope that we have of survival as a nation with a reasonable standard of living depends upon whether we have sense enough as free individuals to harness quickly to our purpose the scientific and engineering ability that we undoubtedly possess. We may not much longer remain free to make this choice if we miss this last opportunity. We require to establish as near full employment of our machines as possible. The introduction of the continuous working week in our steel plants has been an outstandingly bold step in the right direction. We must endeavour to follow that lead in other directions. We cannot afford the luxury of any class not working, individually or as a team, at full throttle and thereby wasting man-power in producing our capital goods and in operating them. We shall probably need to increase our average hours of work, not decrease them.

We must achieve increased mechanisation of our processes, particularly by way of introduction of high capacity mass production units, which will involve concentration of output. There must be no restriction in the development and use of equipment which will improve efficiency in any direction. It is worth while looking back over the last fifty years and reflecting that the steady rise in the standard of living was mainly due to a constant increase in productivity. This advance was largely achieved by increased mechanisation.

"The great problem to-day" said a trade union leader recently, "is to get men to change ideas that are fixed, and which were acquired in an entirely different set of industrial circumstances. These ideas and habits of thought had some relevance and some meaning in the social and industrial conditions which existed in our fathers' time, but to-day are out of date, useless and, indeed, dangerous to our productive effort on which, in the last resort, our standard of living depends." This change of attitude is necessary to our survival. If that change is made quickly Mr. Judge believed we would not only survive as an industrial nation, but as individuals we would advance to an even fuller standard of living. We have the technical skill and the mechanical resources to do this, but we have yet to acquire and develop the attitude of mind which will give them full play.

Original Whittle Engines now Museum Pieces

IT is rather remarkable that pioneer work carried out and developed to less than a decade ago should now find a place in a museum. We are so apt to regard museum exhibits as representatives of past generations and tend to overlook the fact that the history of engineering in particular is continually being developed.

This is emphasised by the recent presentation to the Nation of the two original Whittle flight engines, which powered the world's first successful jet-propelled aircraft, the WI and the WIX, which have now become museum pieces ; the former in the Science Museum and the latter in the Smithsonian Institution, Washington.

The WI powered the Gloster-Whittle E28/39 on its first official flight at R.A.F. Cranwell on May 15th, 1941. The sister engine, WIX, built as a test-bench engine, was actually the first to become airborne in an unofficial flight in March, 1941. Both these engines were designed by Sir Frank Whittle—at that time a Flight-Lieutenant in the R.A.F.—and built for Whittle's company, Power Jets, Ltd., by the British Thomson-Houston Company, Ltd., Rugby. From these engines have developed the series of centrifugal types in regular use to-day, including Britain's latest bid for supremacy in the passenger-transport field—the De Havilland Comet. At the request of the United States Government, the WIX engine was lent to the General Electric Company, Lynn, Massachusetts, for study purposes in October, 1941, accompanied by British engineers and full information on British progress in the gas turbine field.

The gift of the engines to the Nation has been made by Power Jet (Research and Development), Ltd., the Government-owned successor to Sir Frank Whittle's original company. They were received on behalf of the Nation by the Rt. Hon. George Russell Strauss, Minister of Supply. It was felt that although WI should remain in the permanent custody of the Science Museum, WIX should return to its second home as a gift from Britain to America. At the ceremony, on October 26th, which commemorated this epoch in aeronautical progress, were Sir Frank Whittle, Mr. Patrick Johnson, one of the earliest of Sir Frank's colleagues and now managing director of Power Jets (Research and Development), Ltd., and the three engineers who accompanied WIX to the United States—Mr. D. N. Walker, Mr. G. B. Bozzoni, who were then working for Power Jets, Ltd., and Warrant Officer J. A. King of the R.A.F.—all three of whom were subsequently decorated.

The presentation of the WIX engine to the Smith-

sonian Institution, on November 8th, marks not only an outstanding development in aeronautical engineering, but will commemorate the technical collaboration between the two countries, which was worthy of the great national alliance which it preceded. Its arrival in the United States on this occasion was public, in contrast to its previous arrival under extremely "hush-hush" conditions in October, 1941, when the possibilities of jet-propelled aircraft were one of the war's most jealously-guarded secrets. The delivery of the WIX at that time, together with a complete dossier on the whole British gas-turbine position, marked the start of one of the most complete phases of Anglo-American co-operation, not only at Government level but between private British and American firms. There can have been few other cases where every detail of such an important development in the technological field as the use of gas-turbines for aero-engines was shared at such an early stage. From this transmission of information developed the American use of the gas-turbine aero-engine.

Pearl Harbour came soon after the British jet unit arrived in America and America's main interest from then on was not that of producing jet-engines for Britain, but of developing the engine for her own use. It was agreed between the two Governments that every scrap of information available from Britain could be used by America but that, after the end of hostilities, America should refrain from making any immediate use of gas-turbine aero-engines until a further agreement had been reached.

This provision was punctiliously honoured by the U.S.A. Within two days of V.J. Day the American Government cabled to Power Jets (Research and Development), Ltd., asking whether they might continue using British patents and technology in the operation and manufacture of aircraft. Permission was readily given since otherwise there would have been a wholesale closing down of American factories and the grounding of many aircraft.

In November, 1945, Patrick Johnson, now managing-director of Power Jets (Research and Development), Ltd., visited the States to negotiate a settlement. As a result the American Government agreed to pay the British Government-owned company \$87,000 dollars (£200,000 at the rate of exchange then prevailing for the use of patented information from 1941 until 1945 and for the use of all un-patented information in perpetuity. Thus closed one aspect of Anglo-American co-operation unique in the history of the war.

Power Jets (Research & Development) Ltd.

Sir Frank Whittle's original company, Power Jets, Ltd., was taken over in 1944 by the Government and renamed Power Jets (Research and Development), Ltd. It operates to-day as a private company under the auspices of the Ministry of Supply, holding some 2,000 British and foreign patents and applications in the gas-turbine field, which it is now responsible for holding and exploiting in the national interest. It also operates the School of Turbine Technology at Lutterworth which seeks to further knowledge of the gas-turbine industry, particularly amongst potential overseas users of gas-turbines manufactured under British patents. The Company has in addition a team of specialist consulting engineers to provide technical advice and help.

Magnetic Saturation Intensities and Curie Temperatures for Some Industrial Permanent Magnet Materials

By L. Ward, M.Sc.

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The work described in this article was undertaken with the object of investigating the phase conditions by magnetic analysis of a representative selection of commercial magnetic materials. The investigation has indicated that, in alloys of the Alnico-type, there is a reversible phase up to the Curie point, and that the cobalt steels, as industrially heat treated, contain mixtures of austenitic and magnetic a phases.

DURING the last few years, mainly because of the great advances in magnet technology and the discovery of the high energy iron-nickel-aluminium-cobalt series of alloys, the importance of permanent magnets has greatly increased. The fundamental causes of ferromagnetism are partly explained but the special conditions of phase relationships and internal atomic structure which give rise to permanent magnetism are still largely a matter for conjecture. The work described in this article was undertaken with the object of investigating the phase conditions by magnetic analysis of a representative selection of commercial magnetic materials. As the field covered was a wide one it was not possible to carry out detailed experiments on each particular material. Such investigations would require numerous measurements on a variety of samples each with slightly different conditions of composition and heat treatment, and therefore, it was decided to cover broadly the whole field of the industrial alloys. The majority of the materials have to undergo several different heat treatment processes in order to develop the optimum properties and the results which were obtained relate only to these particular treatments.

Apparatus and Experimental Details

The experimental procedure was to investigate the conditions at magnetic saturation; this eliminated any effects of the value of the magnetising field on the induced intensity of magnetisation.

An apparatus which is particularly suitable for this work is the magnetic balance due to Sucksmith,¹ a brief description of which is given below.

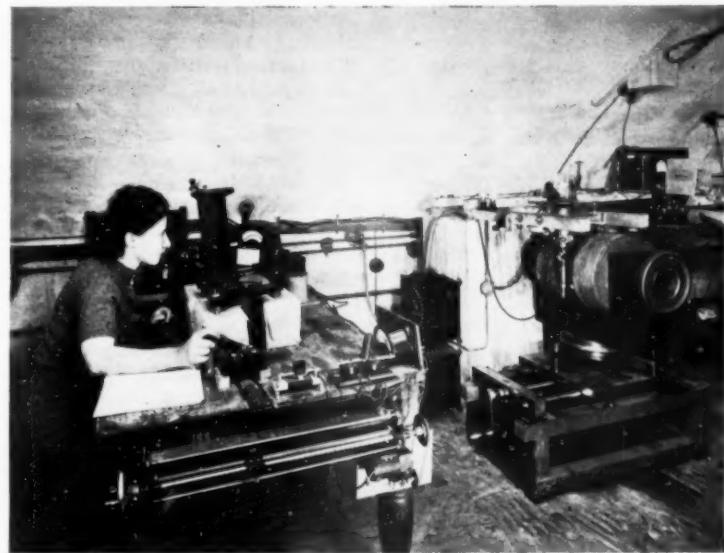


Fig. 1.—General view of Sucksmith balance.

The general lay-out of the apparatus is shown in the photograph (Fig. 1). A ferro-magnetic body situated in a non-uniform magnetic field is subjected to a force proportional to $m \sigma \frac{dH}{dx}$ where m is the mass and σ

the saturation intensity, defined as the saturation intensity of magnetisation divided by the physical density. In the Sucksmith balance this force is balanced against the restoring force in a thin ring, small distortions of the ring being measured by means of an optical lever system (Fig. 2). The specimen under investigation, B , is fastened to the end of a stiff molybdenum rod, the other end of which is rigidly attached to the lowest part of the ring. The ring, which is 2 in. in diameter, is made of copper-beryllium strip about $\frac{1}{4}$ in. wide and 0.015 in.

thick, and is suspended so that the rod hangs vertically. Two small brass tubes are soldered to the ring to hold the brass "flags" to which the mirrors M are attached by shellac. The ring system is mounted on a substantial brass base. In order to maintain the rod in a vertical position against the magnetic forces in the horizontal direction, two flat spiral springs S are placed at points along the rod. These springs, although they offer very high resistance to any movement in a horizontal plane, have a negligible resistance to vertical movement and do not affect the characteristics of the ring. The system is highly susceptible to vibration and several precautions are necessary to prevent the ring from shaking during use. The base plate is firmly bolted to thick wooden beams projecting from a concrete pillar built

¹ Sucksmith. *Proc. Roy. Soc., A.943, Vol. 170, pp. 551-569, April, 1939.*

up from the floor. In addition, oil damping is provided in the form of a light vane screwed to the ring and dipping into a small pot of thin oil. The combination of these methods serves adequately to prevent any disturbance due to vibration.

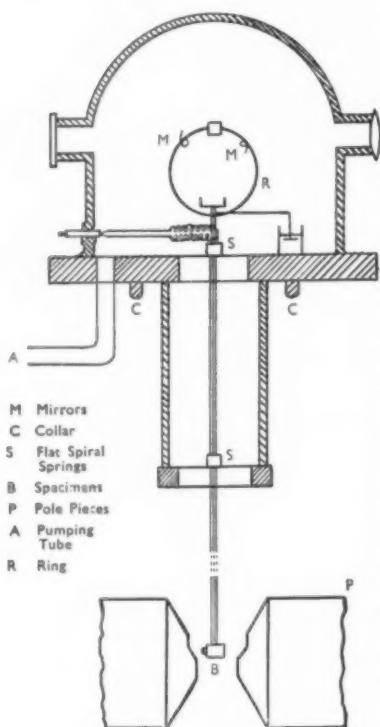


Fig. 2.—Details of Sucksmith balance.

The ring system is covered by a glass dome with two diametrically opposite ports, one of which is sealed by a 50-cm. focus lens and the other by a piece of plane glass. The dome has a ground base so that vacuum-tight joints can be made.

The specimen under investigation, measuring about 1 mm. by 4 mm., is enclosed in a small platinum-iridium box. This fits into an eccentric hole in a small block of similar material screwed to the lower end of the molybdenum rod. Platinum-iridium is chosen because repeated heating does not alter appreciably its magnetic or mechanical properties. The temperature of the specimen is measured by a thermocouple located in a slot in the block, the leads being brought out up the rod and insulated from it by twin-bore silica tubing.

A beam of light from a source box passes through the lens, is reflected in turn from each of the mirrors and is

focused on the cross-wires of a vertical travelling microscope placed about 1 metre away. The light source consists of a multi-filament lamp in a brass box fitted with an adjustable slit aperture, and by rotating the lamp it is possible to select the best filament. When the field is switched on, the ring is slightly distorted, and the light image is deflected.

The specimen is located in the gap of an electromagnet having specially designed pole-pieces. The dimensions of the specimens indicate that a high demagnetising factor is to be expected, and to obtain complete saturation a field of about 15,000 oersteds is required. A steep field gradient is also necessary in order to produce a large deflection. Both these conditions are fulfilled by having stepped pole-pieces. The vertical distribution of force in the magnet gap has a maximum value at a certain point at which position it is desirable to operate. The rigidity of the system ensures that this same point is repeated following a displacement.

The characteristics of the balance may be determined by adding weights to a small pan attached to the ring. It is found that, up to large deflections, the deflection of the light image is directly proportional to the force. If the deflection can be kept within this range, and if the specimen does not move into a region where the field gradient has changed, both conditions which usually obtain in practice, it can be assumed that the deflection is proportional to the intensity.

Although it is possible to measure absolute values of the saturation intensity by evaluating the restoring force in the ring, it is more usual to express the results as relative to pure iron, which has an intensity σ at saturation of 217.8, where $\sigma = \frac{J_s}{\rho}$.

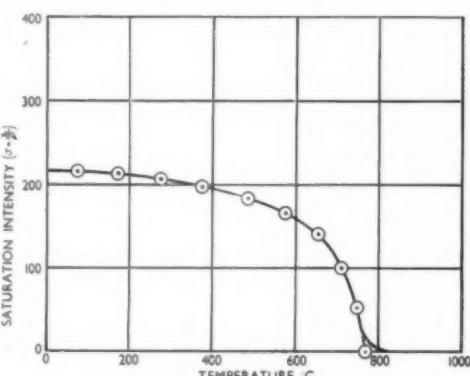


Fig. 3.—Saturation intensity-temperature curve for iron (body centred cubic phase).

For measurements at high temperatures, the specimen is enclosed in a furnace attachment consisting of a quartz tube, wound with Nichrome tape thermally insulated by asbestos cement, and sealed into a brass envelope. The envelope fits over the collar C, the whole system being operated in a vacuum at high temperatures. Measurements of intensity are taken at various temperatures during the heating and cooling operations.

Sucksmith and his collaborator have used this balance for investigations on nickel-iron^{2,3} and nickel-iron-aluminium⁴ systems, and further details on experimental methods may be obtained from their publications.

Measurements of Magnetic Materials

Having described the apparatus, the account of the main experimental work can be commenced. The normal intensity-temperature curve for a metal or alloy consisting of a single magnetic phase is shown in Fig. 3, and, it will be seen, is perfectly smooth and reversible. When more than one magnetic phase is present, each will have its own Curie temperature and the curve will show the Curie temperatures of all the constituents as points of inflection. The magnetic materials investigated were those used in the manufacture of permanent magnets, and included the more recent iron-nickel-aluminium-cobalt alloys—the Alnico alloys and Aleomax II—as well as the older cobalt-, chromium- and tungsten-steels. In addition to a general series of measurements on the heat-treated samples, a special investigation was carried out on 35% cobalt steel and this will be described later.

The specimens used were in two forms. The cobalt-, chromium- and tungsten-steels, although hard, are

fairly tough and it was possible to grind roughly cylindrical specimens about 1 mm. in diameter and 4 mm. long, the exact shape being unimportant provided the applied field was sufficient to produce saturation. The Alnico type of alloys presented a different problem. Their crystal size is very large, and the alloys are exceedingly brittle so that the grinding of small

² Pickles and Sucksmith. *Proc. Roy. Soc. A*, 1962, Vol. 175, pp. 331-344, May, 1940.

³ Hoseitz and Sucksmith. *Proc. Roy. Soc. A*, Vol. 181, pp. 303-313, 1943.

⁴ Sucksmith. *Proc. Roy. Soc. A*, 1947, Vol. 171, pp. 524-540, July, 1939.

specimens is difficult. The practice adopted was to crush the sample and select a number of small fragments with which the specimen box was filled. Sucksmith demonstrated that coarse powders gave identical results to those obtained from solid specimens. In the first instance, all the specimens were taken from materials in the final heat-treated condition.

Table I shows the results obtained on the materials studied together with the composition by weight and the magnetic characteristics.

Curves of saturation intensity plotted against temperature for these materials are shown in Figs. 4, 5 and 6. The variation of density with temperature is small and can be neglected.

It is possible to divide the materials into two main series—the Alnico series and the cobalt steel series. The Alnico alloys each contain the same elements, the main differences being in the nickel contents. Similarly the main variable in the composition of the cobalt steels is the cobalt content.

Behaviour of Iron-Nickel-Aluminium-Cobalt Alloys

The room temperature saturation intensities for the Alnico alloys show little difference except a slight decrease in saturation intensity with an increase in nickel content of the order of magnitude to be expected. Similarly, the Curie temperatures show very little variation and the intensity curves generally indicate a purely reversible single magnetic state.

The heating curve for Alcomax II is smooth and gives a Curie temperature of 830° C. The cooling curve follows

a path lower than that of the heating curve and at about 400° C. there is a slight change in curvature. This indicates that at the high temperature the original phase has been partially transformed into a phase with a Curie temperature of 400° C. Alni gives purely reversible curves and in this respect is similar to Alnico.

In connection with the shape of these curves the investigations of W. Zumbusch⁵ are interesting. Zumbusch studied photomicrographs of an anisotropic permanent magnet alloy of similar composition to Alcomax. Sections taken from samples subjected to various quenching treatments up to 1,300°C., showed, on examination, that below 930° C. and above 1,200° C., the quenched alloy was in a homogeneous state, which is presumably a solid solution. On the other hand, quenching from between these temperatures produced a mixture of two constituents giving a martensite-like structure. This conclusion is in agreement with X-ray results and also

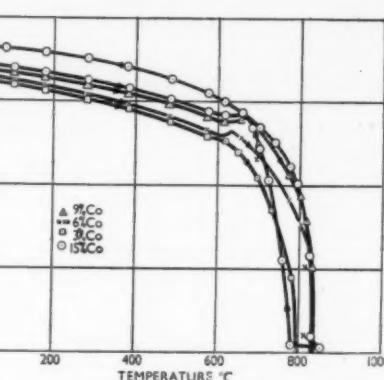


Fig. 5.—Saturation intensity-temperature curves for cobalt steels.

Fig. 4.

—Saturation intensity-temperature curves for iron-nickel-aluminium alloys.

magnetic tests on bulk samples, being represented by a decrease in available magnetic energy. In commercial production Alcomax II is quenched by air-cooling from 1,200° C., so that the homogeneous state would be expected. Heating beyond the Curie temperature should produce the martensite-like state, one component of which is probably that with the Curie temperature of 400° C. It seems possible that the homogeneous condition really comprises two or more distinct phases but it was impossible to detect these individually, probably because the

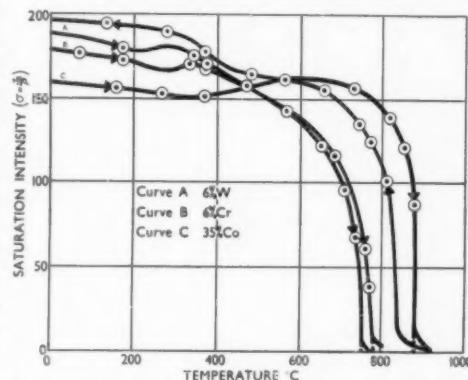


Fig. 6.—Saturation intensity-temperature curves for 35% Co, 6% W, and 6% Cr steels.

⁵ Zumbusch. *Archiv. für das Eisenhüttenwesen*, Vol. 16, pp. 101-112, Sept., 1912.

TABLE I.—MATERIALS STUDIED, THEIR COMPOSITION BY WEIGHT AND THEIR MAGNETIC CHARACTERISTICS

Magnetic Material	Nominal Composition (Wt. per cent.) (Fe balance)								Brem gauss censored	H _o gauss censored	(BH) _{max} x 10 ⁻³ E./cc.	J _s	Specific Saturation Intensity $\sigma = \frac{J_0}{p}$	Curie Temp. °C.	
	C	Ni	Al	Co	Cu	W	Cr	Mo							
Iron	—	—	—	—	—	—	—	—	—	—	—	7.87	2,700	218	76 ₁
Alnico I Alloy	0.06	18	10	13	6	—	—	—	7,300	500	1.6	7.45	900	133	80 ₁
Alnico II Alloy	0.06	—	10	12	6	—	—	—	8,000	350	1.6	7.45	1,010	136	79 ₁
Alnico III Alloy	0.06	20	10	12	6	—	—	—	6,000	600	1.6	7.60	980	131	79 ₁
Alcomax II Alloy	0.06	11	8	21	4	—	—	—	12,500	550	4.3	7.55	1,215	161	83 ₁
Alni Alloy	0.06	25	13	—	4	—	—	—	6,000	500	1.3	7.00	785	112	71 ₁
35% Cobalt Steel	0.85	—	—	35	—	4	6	—	9,000	250	0.96	8.15	1,300	160	88 ₁
15% Cobalt Steel	1.05	—	—	15	—	—	9	1.5	8,300	180	0.62	7.90	1,375	174	82 ₁
9% Cobalt Steel	1.05	—	—	9	—	—	9	1.5	8,000	160	0.54	7.80	1,340	172	82 ₁
6% Cobalt Steel	1.05	—	—	6	—	—	9	1.5	7,500	135	0.43	7.75	1,285	166	82 ₁
3% Cobalt Steel	1.05	—	—	3	—	—	9	1.5	7,200	125	0.4	7.70	1,260	164	79 ₁
6% Chromium Steel	1.05	—	—	—	—	—	6	—	9,500	65	0.30	7.75	1,400	181	79 ₁
6% Tungsten Steel	0.67	—	—	—	—	6	0.4	—	10,600	65	0.32	8.10	1,530	189	76 ₁

Curie points of the phases are nearly the same.

Magnetic State Behaviour of Cobalt Steels

The cobalt steels may be treated as a group in which the composition varies only as regards the cobalt content; the steels also contain chromium, tungsten and molybdenum. The intensity-temperature curves are shown in Fig. 5. The room temperature saturation intensities show a general increase with increasing cobalt content. Stoner⁶ gives diagrams of the saturation intensity against composition for cobalt-iron alloys. Naturally the presence of the other elements will have an influence on the constitution but for low percentages of cobalt (up to 40%), there is a range of solid solutions with high saturation intensities, the intensity increasing with the cobalt content.

The Curie temperatures of these steels show a general decrease with decreasing cobalt content. This is in agreement with expectations bearing in mind the high Curie point of pure cobalt (1,150° C.). The intensity-temperature curves for all these steels are interesting. On heating, the intensity falls steadily up to a temperature of about 600° C. At this point, the intensity begins to rise reaching a maximum value at 630° C., from where a smooth and rapid fall is observed up to the Curie temperature. On cooling, the specimen does not begin to regain its ferromagnetism until its temperature is 40°–50° C. below the Curie point; then a rapid rise in intensity occurs and the curve rejoins the heating curve, passing through the maximum at 630° C., and continuing smoothly to a room temperature intensity some 10% higher than the original value. Re-heating causes the

saturation intensity to follow the cooling curve to 630° C., and then to trace the previous heating curve up to the Curie temperature. The hysteresis effect at the Curie temperature is usually associated with a phase change which needs some time for its completion.

The hysteresis effects are most marked in the case of the 35% cobalt steel. Fig. 6 shows the heating and cooling curves for this material. A more detailed investigation into the constitution of 35% cobalt is described below.

The curves for the chromium and tungsten magnet steels show similar effects to the cobalt steels. The temperatures at which the increase in intensity occurs are much lower than the corresponding ones for the cobalt series; saturation intensities are high and Curie temperatures low.

Behaviour of 35% Cobalt Steel

In an attempt to obtain more information about this increase in intensity at comparatively high temperatures, the 35% cobalt steel was chosen as the effect was most pronounced with this material. A number of small specimens were made from the same sample of steel, the heat-treatment was carried out *in vacuo* in small diameter quartz tubes which could be plunged into cold water without danger of cracking. The experimental procedure was to soak each specimen at a given temperature for a period not less than 24 hours and, in the case of the lower temperature heat-treatments, for periods up to 48 hours. The temperature of the treating furnace was constant to about ± 5 ° C. The specimens were quenched in cold water immediately on withdrawal from the furnace. The room temperature saturation intensities and the intensity-temperature curves were determined

by the Sucksmith balance method described earlier. The room temperature saturation intensities plotted against the quenching temperatures are shown in Fig. 7, while the intensity-temperature curves for specimens quenched between 300° C. and 550° C. are plotted in Fig. 8. It will be seen that the bump on these curves tends to be concentrated towards the higher temperatures as the quenching temperature is increased and at 550° C. it disappears altogether. This suggests that by quenching from this temperature, a single magnetic constituent is produced.

From the work of Matthews⁷ and of Sykes⁸ it appears that, in alloys containing iron, tungsten and cobalt in approximately the proportions found in the 35% cobalt magnet steel, there are at least two solid solution states which may be produced by heat treatment. Above 950° C. there is a face centred cubic solid solution (γ). This is subject to grain refinement during rapid cooling from above 950° C. in much the same way as austenite in steel. Owing to the low diffusion rate of tungsten, however, there is no precipitation of a tungsten rich phase until the alloy is re-heated to at least 300° C. Isothermal decomposition at temperatures below 950° C. gives a "pearlitic" structure. Matthews found that the austenite-martensite transformation is not complete in magnet steels and that there was usually 5–10% of austenite in the finished steel. He suggested that this residual austenite was a cause of increased permanence.

On the basis of two constituents the results obtained with 35% cobalt steel may be interpreted. In a pure binary system γ would be stable at high temperatures, and would normally be transformed into martensite α on

quenching. The presence of other elements in the steel, however, may slow up the rate of transformation so that a considerable percentage of γ may be retained at room temperature. On re-heating the steel the saturation intensity of the α -part will fall in the usual manner until a temperature is

⁶ Stoner, *Magnetism and Matter*, pp. 532–3.

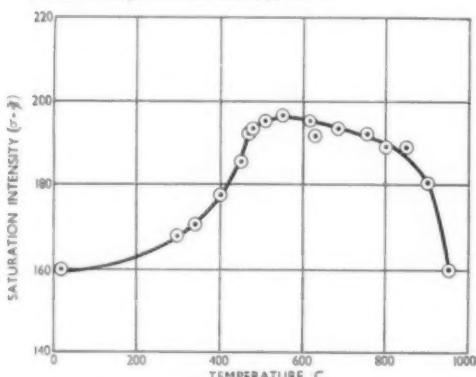


Fig. 7.—Saturation intensity-quenching temperature curve for 35% Co steel.

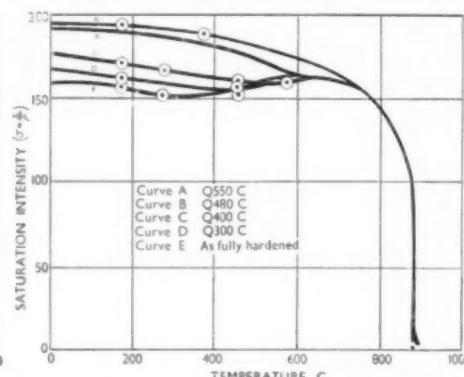


Fig. 8.—Saturation intensity-temperature curves for 35% Co steel with different heat treatments.

⁷ Matthews, *A.S.S.T.*, **8**, 565, 1925.

⁸ Sykes, *A.S.M.*, **25**, 953, 1937.

reached when the γ - α transformation rate is speeded up. At this point a rise in saturation intensity would be expected, and finally the intensity will then decline until the Curie point is reached. Requenching the finished steel should transform a portion of the γ into α and so give rise to an increase in room temperature saturation intensity.

If this is correct it will be seen that normally-treated 35% cobalt steel contains about 20% of γ . Matthews found 5-10%. Magnetic energy tests on bulk samples containing almost pure α show a marked decline in available energy $(BH)_{max}$. Folk⁹ shows that

there is a critical cooling rate for 35% cobalt magnet steel and suggests quenching from different temperatures for magnets of different mass.

Summary

An investigation into the magnetic phases present in some permanent magnet steels and alloys has indicated the following points:

- (a) In alloys of the Alnico-type there is a reversible phase up to the Curie point.
- (b) The cobalt steels, as industrially heat-treated, contain mixtures of "austenitic" and magnetic α phases. Heating causes the austenitic phase to be converted

to the α body-centred cubic phase. The presence of a small percentage of non-magnetic austenite in the steels would seem to be normal in magnets having maximum magnetic energy.

Acknowledgment

The work described above was carried out at the B.S.A. Group Research Centre, Sheffield, and the author wishes to thank the Director, Mr. D. A. Oliver, M.Sc., F.I.M., F.Inst.P., for his interest, and for permission to publish this article. Thanks are also due to Dr. K. Hoselitz, F.Inst.P., and to Mr. D. Hadfield for a number of useful discussions.

⁹ Folk, A.S.M., **37**, 192, 1946.

Corrosion and Corrosion Fatigue of Steel

A joint meeting of the Iron and Steel Institute and the British Iron and Steel Research Association was held at the Institute's offices in London on October 18th, 1949, to discuss several papers on corrosion and corrosion fatigue of steel. This report of the meeting, which was presided over by Mr. T. M. Herbert, chairman of the Corrosion Committee of the Research Association, presents some of the main features discussed.

CORROSION, which takes such a heavy toll on the service and performance of most industrial metals, can be reduced effectively by the application of science and technology, but efforts in this direction are really only on the fringe of the problem. Discussions on the subject not only emphasise its importance but indicate the tremendous nature of the task of reducing its effect in service conditions. Each discussion reveals some aspect of the subject which assists in appreciating more fully the difficulties involved. A recent discussion on the subject at a joint meeting of the Iron and Steel Institute and the British Iron and Steel Research Association was such an occasion. There were two sessions; four papers being presented for discussion at the first and four at the second. In the following notes are given brief summaries of papers and discussions in which important aspects are emphasised.

A Simple Form of Accelerated Atmospheric Corrosion Test

The four papers presented at the first session were concerned with atmospheric corrosion, the first of which, that on the above subject, was presented by R. ST. J. PRESTON of the Chemical Research Laboratory, Teddington. Introducing the subject, Mr. Preston emphasised that this was not an accelerated weathering test, but one where the main agents of corrosion were produced in controlled conditions. He described how specimens are subjected to corrosion in a warm humid atmosphere containing sulphur dioxide. The experimental work was carried out with steam-heated apparatus, since replaced by a thermostatically-controlled electrically heated type, of simple construction.

The effects of temperature and concentration of sulphur dioxide on rates of corrosion of bare and

phosphated steel, with and without paint coverings, had been examined, and it had been found that corrosion rates increased with rise in temperature and with increase in sulphur-dioxide concentration. Pre-treatment by phosphating improved the corrosion resistance of painted steel, particularly when the paint coating was less than 0.5 mil. thick. A thick phosphate coating, which completely covered irregularities in the steel surface and therefore did not conduct electricity showed a resistance in outdoor exposure tests much superior to that of bare steel. Results obtained with this apparatus were far more representative of the behaviour of protective material in industrial atmosphere than were those given by the salt spray test.

In the discussion, MR. S. C. BRITTON agreed that accelerated corrosion tests, although always having limitations, were a necessary evil. Tests giving rapid and reproducible results were needed both by users of protective processes and by the developers of new materials. Both classes should welcome an addition to the commonly used salt-spray test which could not reflect all conditions of service. For those applying protective processes for use in industrial atmospheres the test as proposed would certainly be useful for checks on the quality of material supplied or for the maintenance of standards on the production line.

Those who wished to choose a new method of protection and those developing new methods might not be so well suited since they wanted an estimate of the useful life of what might be rather different materials. He was not convinced that the method as it stood could give even a reliable order of merit for dissimilar materials. He recalled tests made with Dr. U. R. Evans in which exposure of different metals over SO_2 solution failed to give orders of merit consistent with those obtained on

atmospheric exposure. He suggested that reliability in predicting the service behaviour of dissimilar materials could be improved, at some sacrifice of acceleration, by using lower concentrations of SO_2 and by introducing into the test periods of drying out of the samples and of washing down with water, both important features of normal corrosion.

MR. T. HENRY TURNER asked whether the test was limited to the standard 6 in. \times 2 in. shape because users require to test irregular shapes such as nuts and bolts. For example, a motor cycle bought during the recent exceptionally dry summer had a number of nuts and bolts which became rusty during the first month's use of the machine and even before they had ever been out in the rain. The makers of this machine were certainly not the worst and took great pains to order suitable components. They would have welcomed a reliable test for the cadmium-plated nuts and bolts which they receive from suppliers. If Mr. Preston's test could be developed for adoption as a British Standard test, that would be of real value to industry, but it seemed undesirable to have to miss out the features of irregular shape and vibration which were experienced in industry.

DR. MAYNE raised the point that this test involved an important principle. Was one attempting to accelerate corrosion or to accelerate the breakdown of paint? Hudson and Banfield had shown how corrosion of a painted steel in a heavy industrial area, such as Sheffield, was not more rapid than elsewhere. It had been thought that the slow results from exposures in Sheffield had been possibly due to the fact that ultra-violet light was kept off the specimens by the atmospheric pollution and by accumulations of deposit, and the effect of ultra-violet light had been confirmed by American work. He pointed out that this factor had been entirely neglected in this test. DR. VERNON said that accelerated tests at Teddington, based on the use of SO_2 , had accurately reproduced the well-known curve showing the effect of copper content on corrosion rate; salt-spray tests had failed to reproduce this relationship.

In reply, MR. PRESTON stressed the importance of controlling the SO_2 concentration. It had been shown by some investigators that at two different SO_2 concentrations the orders of resistance to breakdown of some materials could be reversed. The influence of ultra-violet light raised by Dr. Mayne in connection with the longer life of specimens exposed in a heavily polluted atmosphere compared with a non-industrial atmosphere, was thought by Mr. Preston to be a matter of shielding of the paint film by the deposit of atmospheric debris. It was not unusual he thought to use black finishing paint, containing opaque pigments for tropical conditions probably with the same objective.

A Study of the Corrosion Resistance of High-Alloy Steels to an Industrial Atmosphere

The next paper, by H. T. SHIRLEY and J. E. TRUMAN, of the Brown-Firth Laboratory, was concerned with this question of industrial atmosphere and its effect on high-alloy steels. It was introduced by Mr. Shirley who described how tests had been carried out which were designed to study the effects of composition and surface finish on the behaviour of higher alloy steels, when exposed for prolonged periods to severely industrial atmospheric conditions, without attention to the cleansing treatment normally recommended for such materials. The intention was to provide information

about compositions for use where regular cleansing would be impracticable, and to compare directly these higher alloy steels with lower alloy steels exposed under similar conditions. There were 450 samples, covering 22 steels and 5 non-ferrous materials, all in sheet form. The three types of surface finish tested were pickled, emered, and mirror-polished. Exposure was for 2 and 5 years with vertical specimens, and for 5 years with specimens exposed at 12° to the horizontal.

The behaviour of 18/8/2½ chromium-nickel-molybdenum and 24/12/3 chromium-nickel-tungsten steels was outstandingly good, with losses of the order of only one-thousandth that of mild steel. In general the more resistant steels tended to show higher losses in the case of the emered samples, than for the other two surface finishes, the effect being small for the vertical exposure stands, but more considerable in the case of the horizontal stands. Although comparison with earlier tests for 1 month and 1 year indicated a general tendency for higher rates of loss in the initial stages of exposure, there was no indication of any notable change in rate attack with time over the 2-year to 5-year periods.

The results for the non-ferrous materials were in line with those expected from previously published figures and show all five materials to give higher losses than most of the special steels. Although in some cases this might be offset to some extent by pitting tendency in the case of the steels, the outstanding superiority of the 18/8/2½ chromium-nickel-molybdenum and 24/12/3 chromium-nickel-tungsten steels was clearly demonstrated, with losses of the order of only a hundredth of those of the non-ferrous materials. He pointed out that the hopes which had long been entertained of the development of a low alloy steel with very high corrosion resistance had faded, though such steels had been developed with, in fact, four times the corrosion resistance of mild steel. It was, therefore, necessary to look to the more highly alloyed steels to find the necessary corrosion resistance for use in severe conditions in spite of their relatively high price.

In the discussion MR. T. HENRY TURNER said that the Atmospheric Corrosion Sub-Committee of which he was Chairman had greatly welcomed Mr. Shirley's data which they regarded as a most valuable contribution to knowledge and industry. Some of the highly alloyed steels might not be "as good as gold" but, they were certainly better than silver as regards corrosion. Moreover they possessed mechanical properties superior to gold or silver. Even the earlier form of 18/8 corrosion resistant nickel-chromium steels had proved of great help. These earlier steels have been used in the United States for Budd-welded corrugated all-steel coaches. The earliest of these trains were now coming into the shops after many years service and the stainless steel was being found to be in perfect condition. However under the very severe conditions of a tunnel atmosphere, these 18/8 alloys had been found to pit rapidly. It was, therefore, of importance that the amazingly good and exciting superior corrosion resistant steels, some of which stood out so clearly in Mr. Shirley's paper, might soon become available and B.I.S.R.A. should take every possible step to make materials of this kind available to industry in this country.

DR. VERNON commented on the remarkably good results obtained from the low-alloy Ni-Cr-Mo steel. Although rusting was not suppressed, the efficiency, weight for weight, of the alloying elements, was even

greater than for the higher-alloy steels, and MR. BRITTON also confirmed that molybdenum additions to chromium-nickel-stainless steels improved their performance on exposure in railway tunnels from 80% of the surface pitted to a few scattered small pits.

The Atmospheric Corrosion of Iron and Steel Wires

This paper by DR. J. C. HUDSON, Head of the B.I.S.R.A. Corrosion Laboratory, described the results of tests on the corrosion of ferrous wires when exposed in an industrial atmosphere for periods of up to 10 years. The rate of corrosion did not vary appreciably with duration of exposure, but was affected by the diameter of the wire, being greater for thin than for thick wires. Certain wrought irons and low-alloy steels proved much more resistant than mild steel. In particular three chromium-copper steels, containing from 0.6 to 0.9% of chromium and 0.5% of copper, showed a remarkable superiority over mild steel after 10 years exposure in Sheffield, having suffered only from one-third-one-half of the loss in weight or tensile strength of the latter.

Dr. Hudson commented that considerable economies would result from the substitution in suitable circumstances of low-alloy steels such as these for mild steel. For example, if it were desired to ensure that a wire installed in an industrial atmosphere had a residual breaking load after ten years of one thousand pounds, it would be necessary to use a diameter of 0.210 in. if the wire were of mild steel but only of 0.149 in. if a chromium-copper steel were used. This would result in a saving in weight of material of 50%. He hoped that there would be contributions to the discussion from representatives of the wire industry dealing with the practicability of making more extensive use of low-alloy steels for fabricated wire products.

During the discussion, MR RICHARDS, MR. BATESON, and MR. BRITTON, all commented that they were fully aware of the superior corrosion-resistance of low-alloy steels as compared with mild steel, but, while the purchasing authorities were naturally keen on the adoption of such materials in cases such as large span bridges, where the use of the higher tensile steel achieved a definite reduction in the initial cost of the structure, it was a different matter to secure their adoption in cases where the resultant initial cost would be greater than that in ordinary mild steel. In the latter cases it was necessary to estimate the ultimate relative total costs of the two types of structure, taking due account of maintenance costs and the longer useful life of the low-alloy steel. These are difficult matters to assess and the same applies to special anti-corrosion methods such as metal-spraying. Every effort is made by enlightened engineers to encourage the use of these superior steels or to secure the adoption of superior anti-corrosion measures, but in general it is extremely difficult and often impossible to persuade the purchaser to look beyond the initial cost of the structure.

MR. T. HENRY TURNER commented that bare steel wire was seldom used. For fencing and signal wires and wire netting hot galvanised or zinc plated were normally specified. The hoist wires or lift cables which might be used bare would be of a higher carbon content and greater tensile strength. The user of a steel wire for fencing or netting would find the bendability of mild steel more convenient than the springiness of higher tensile steels, and if corrosion resistance were provided by zinc

they would have a more pleasing appearance than a rusty low-alloy steel.

The superiority in corrosion resistance of zinc might be 18 times that of mild steel and low-alloy steels were not that much better than mild steel. A uniform thick coating of electro-deposited zinc in place of the old blobby hot galvanised coating would probably be more popular with engineers than low-alloy steels and if the trade did not produce adequately corrosion-protected steel wire, tennis court wire netting and other wire fencing would probably be found to be made of aluminium in ten years time.

In reply to a question by DR. VERNON, DR. HUDSON said that there had been no pitting observed on the low alloy wires exposed, but only a surface roughness on the undersides, where raindrops hang.

Climatic Effects on the Corrosion of Steel

MR. J. DEARDEN, the chief metallurgist at the Scientific Research Department of the British Railways, London Midland Region, introducing his paper, said, exposure of specimens to the atmosphere in Derby over a number of years had been used in an attempt made to correlate the corrosion of steel in a moderately industrial atmosphere with the hours of rainfall registered by a recording rain gauge. No such relation was found, as only 35-40% of the total corrosion occurred during the hours when the rainfall was sufficient to register on the gauge. A further 35-40% was due to the effects of humidity, and the balance is presumed to have occurred during periods of drizzle and when the specimens remained wet after recordable rainfall had ceased.

DR. VERNON thought that the method adopted by Mr. Dearden had over-emphasised corrosion due to rain and had correspondingly vitiated his estimate of the relative contributions of other weather factors. In industrial atmospheres rain is largely beneficial in washing off corrosive deposits.

MR. T. HENRY TURNER welcomed the paper because much work was needed on micro-meteorology. The liability to corrosion must vary from street corner to street corner and not merely from country to country, depending on the locality of sulphur dioxide emissions from power houses and soot from houses. Mr. Dearden's specimens sheltered from rain would also be sheltered from soot and grit deposits. We have evidence that corrosion may vary from yard to yard along a tunnel depending on the location of ventilation shafts. We regularly find the backs of large steel specimens exposed at 45° to the horizontal to be in a bad condition as compared with the top which, although exposed to the rain, was also dried by the sun and wind. Condensation is sometimes a more important source of corrosion than rain.

DR. HUDSON also touched on this point saying that appreciable rusting of steel only became possible when the relative humidity reached 80%. The actual severity of rusting then depended upon the degree of atmospheric pollution present.

The Effect of Shot-Peening Upon the Corrosion Fatigue of a High Carbon Steel

The four papers presented in the second session were concerned with corrosion fatigue and protective coatings, the first of which, on the above subject, was introduced by DR. U. R. EVANS, who emphasised that improved fatigue properties resulting from shot-peening had long

been realised, but its effect on corrosion fatigue was less well known. The object of the research described was to provide information on that matter. He emphasised that the type of peening must be specified, and carried out under controlled conditions. The paper described how steel peened in seven different ways had been subjected to corrosion-fatigue in very dilute sulphuric acid and in seawater; fine-ground unpeened steel was tested for comparison. It was found that peening greatly increased the endurance at stress ranges commonly prevailing in service. The addition of sodium carbonate and sodium bicarbonate to sodium chloride, however, diminished the endurance of peened specimens, probably by localising the action; at certain alkali contents, peened specimens had a shorter life than finely-ground specimens.

Corrosion-Fatigue of Steel under Asymmetric Stress in Sea Water

DR. A. J. GOULD of Sheffield University then introduced the above paper in which he stated that the stresses—"locked up" or "residual"—in the plates of welded ships, added to the stresses of propulsion, present problems of corrosion fatigue. Most laboratory tests have been carried out on rotating beam machines which appear to predict much shorter working lives for many structures subjected to alternating stress than would actually be realised in service. Using a 6-ton Haigh push-pull electro-magnetic machine, Dr. Gould was able to use test pieces of diameter not less than $\frac{1}{2}$ in., which this machine could, subject to push-pull forces of nearly constant intensity over the whole of their cross sectional area, the stress being "fluctuating" rather than "reversed."

The severity of corrosion fatigue under reversed stress with superimposed tensile stress in sea water was found to depend on the stress-range, but was almost independent of the mean stress of a cycle, provided that this was not excessive. The result was obtained on polished specimens, scale-covered specimens, and specimens descaled by pickling. The endurance as measured on a push-pull machine was roughly five times as great as that measured on a rotating-beam machine for the same materials and stresses. In well-aerated sea water, the test-piece broke at its most slender section. In the absence of aeration, fracture usually occurred about 1 cm. below the water-line, even though the stress here was not at its maximum.

In the joint discussion on these two papers comments from MR. FORBES were read, in his absence. He thought that corrosion fatigue failures arose more often in ships than was realised. Long periods between repaints, with continual vibration allied to "panting" or local bending action could occur. Corrosion fatigue had, in fact, caused actual pieces of the hull near the bows of a high-speed vessel to fall out. He was of the opinion that more work on these lines was needed since the present results, though valuable, were limited in scope.

MR. A. J. T. GIBBONS, who read Mr. Forbes' remarks, thought that the locked-in stresses which occurred due to restraint in a welded structure, were usually small, of the order of a few tons per square inch, in the "body" of the plate. The residual stresses near the weld, however, were much larger, though self-balancing, and could occur even without excessive restraint. These facts should be kept in mind when planning future work.

MR. A. J. BROWN commented on shot peening. He gave some interesting results of experiments on Si-Mn steels to ascertain the relative effects of normal shot blowing for descaling, shot peening, and protection of the surface of the test-pieces against corrosion, in connection with the adoption of torsion bars for front-end suspension motor cars. The test pieces had an effective diameter of $3/16$ in. Corrosive influences were obtained by maintaining a meniscus of tap water along the length of the specimen. It appeared that the best results were given by specimens ground, shot-blown and with two coats of paint, and it was concluded that there was a possibility of "over-peening" such small sections.

A surprising result was the fact that specimens ground, shot-blown, phosphate treated and given two coats of paint gave less good results than specimens ground, shot-blown and given two coats of paint. A possible explanation was that with unsealed phosphate films the corroding medium was uniformly absorbed on the surface of the specimens, and localised attack occurred less readily, whereas when the phosphate coating was sealed with paint, local failure of the paint film would accelerate corrosion-fatigue by localisation of the attack due to the great adhesion of the coating preventing spread of the corrosion effects beneath the protective paint.

High-Speed Rotor Tests of Paints for Under-Water Service.

DR. WORMWELL introduced this paper and referred to an apparatus which had originally been devised at the Chemical Research Laboratory to investigate the behaviour of metals moved at controlled speeds through corrosive liquids. In this apparatus an ebonite holder carried suitably prepared metal specimens, rotated at a controlled speed, the nature and amount of corrosion after varying periods of test being estimated by measurements of weight-loss and depth of corrosion penetration, supplemented by visual examination. The apparatus lent itself to modifications for testing specimens of different forms, for example, rings cut from tubing or portions of wire cable.

A series of 50-day tests of typical ships'-bottom compositions in artificial sea water showed good correlation with raft tests on the same compositions which had extended over 29 weeks. The rotor test could be taken to afford a rapid and convenient method of assessing the value of paint systems intended for use on submerged metals.

During the war when it was necessary to devise an accelerated method for testing anti-corrosive paints, it was found that the apparatus simulated conditions near the hull of the ship. Recently, tests on some of Fancutt and Hudson's formulations had been carried out and although the order of merit was not identical in all respects in the laboratory and raft tests, paint No. 32 (pigmented with white lead, basic lead sulphate, and Burntisland red in a modified phenol-formaldehyde resin-oil medium) showed outstanding superiority in both series. The influence of modifications in the medium of paint No. 173 (based on No. 32) was studied in a further series of tests: some differences were observed, although all the paints showed good adhesion.

The apparatus had demonstrated a rapid and reliable method of showing up defects and arriving at an order of merit for different paints under immersed conditions.

Continued on Page 30

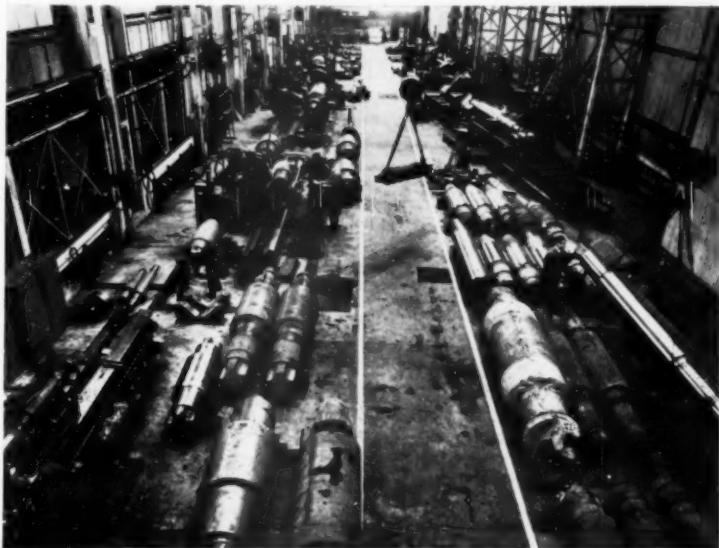
Capital Equipment for Metal Working

Extrusion and Rolling Mill Plant

Post-war requirements in the metal working industries have emphasised the need for heavy plant. Recently the Loewy Engineering Co., Ltd., and F. B. Engineers, Ltd., held "open-house" at the works of the latter Company, when an opportunity was provided for visitors to see the facilities available for meeting this demand.

THE Loewy Engineering Company, Ltd., has, during the last thirteen years, established for itself a high reputation in the heavy press and rolling mill fields. As readers will be aware, the Company specialises in hydraulic presses and power systems, hot and cold rolling mills and associated auxiliary equipment for the steel and non-ferrous metal industries.

F. B. Engineers, Ltd., whose works are at Carlisle Street, Sheffield, is an associated company of Thos. Firth and John Brown, Ltd. An arrangement between F. B. Engineers, Ltd., and the Loewy Engineering Co., Ltd., fulfils the need for extensive facilities for building the heaviest rolling mill and hydraulic press equipment, which is designed to meet the trend of post-war requirements. Light Machines, Ltd., a joint undertaking between Loewy and Firth-Brown, has been established at

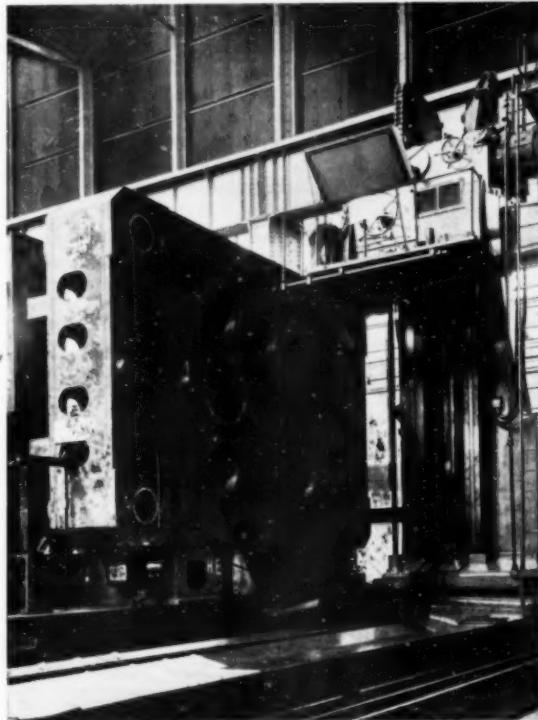


A view of the roll shop showing some finished rolls.

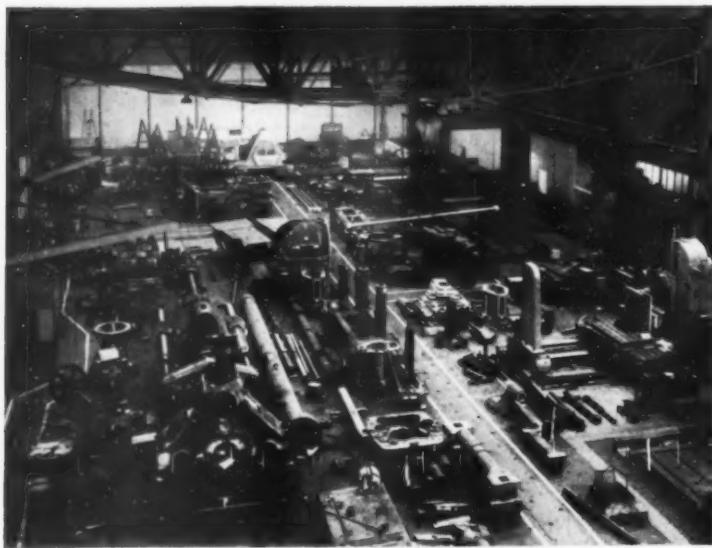
Yeovil, Somerset, to manufacture specialised precision equipment for Loewy hydraulic machinery and prototypes of new equipment.

The foregoing arrangements have proved eminently satisfactory and, with a view to showing the facilities available to those in the metal working industries who are concerned with rolling and extrusion, the two companies recently invited some 200 guests to visit the F. B. Engineers' works at Sheffield. The Yeovil works of Light Machines, Ltd., were also open for inspection the same week. The guests included the Master Cutler, Mr. W. R. S. Stephenson and the Hon. R. A. Balfour, President of the Sheffield Chamber of Commerce, whilst some fifty visitors from overseas represented Companies from Sweden, Belgium, France, Luxembourg, Switzerland and Spain. Attending to the guests were Sir Arthur Matthews (Managing-Director), Mr. E. Mensforth (Deputy Managing-Director), Mr. S. W. Rawson (Director), Mr. J. E. Owston (Director), and Dr. C. Sykes (Director of Research) for Firth-Brown, and Mr. K. Guttenstein (Managing Director) Mr. E. K. L. Haffner (Director) and Mr. M. Langen (Director) for the Loewy organisation.

The Loewy Engineering Co., Ltd., has, since its formation, specialised in the design and manufacture of "Tailor-made" plant, each project being designed and built in accordance with the individual requirements of the customer. During the course of the visit, a large range of such plant was seen going through the shops during machining and in course of erection. This included a complete 40 in. blooming mill; a 4-stand



Upper platten weldment for a 2,500-ton flanging press on a horizontal boring machine.



General view of one of the engineering shops. Bottom left is a Loewy 2,500-ton extrusion press.

34 in. 2-high structural mill, complete with hot and cold finishing end and including the heaviest roller straightener built to date in this country; a complete continuous hot strip mill with four 2-high roughing stands, six 3-high finishing stands, one horizontal and three vertical edging mills, including flying shear, horizontal coilers, etc.; a 20½ in. and 55 in. × 88 in. cold strip mill; 3 extrusion presses for tube, rod and strip manufacture (with pressure water station); a double-acting, 1,500/1,000-ton flanging press, with blank holder (with pressure water station); a 3-stage 450-ton flanging press; a 600-ton triple-ram scrap baling press (a self-contained unit with direct pump drive); 2 forging presses (with water pressure station); 6 heavy and 4 medium-heavy asbestos cement presses (each representing a self-contained unit with direct pump drive); and a weldment for a metal powder press (the actual press is manufactured at Yeovil).

F. B. Engineers, Ltd.

The works of F. B. Engineers, Ltd., have been laid out and equipped since 1947, their general conception being

that of a self-contained unit specialising in the manufacture of exceptionally large metal working and similar equipment. The shops are conveniently placed with regard to both road and rail transport for the handling of heavy and bulky components. They are located in a district where it is possible to obtain, economically and expeditiously, large steel forgings, heavy iron and steel castings and the necessary specialist engineering services, e.g., the cutting of large gears. Eighteen electric overhead cranes and many auxiliary jib cranes serve the works. These include Arrol, Adamson, Craven and Morris units of 100, 75, 70, 50-ton and smaller sizes with, in addition, a universal coupling beam enabling exceptional lifts up to 160 tons to be handled.

The shops comprise a fabricating and welding department, machining and erection units and the necessary ancillary services. The whole equipment has been designed to be reasonably self-contained; at the same time, however, the Company is able to draw on the specialised and exceptional resources of associated Companies which include such machine tools as lathes up to 100 ft. between centres, swinging 15 ft. 8 in. in diameter and a vertical boring mill taking up to 40 ft. in diameter.

Fabrication

Fabrication facilities for the production of weldments are mostly in two bays, under cranes up to 50-tons capacity, whilst provision is made for the final welding of structures up to 160-tons in weight in an adjacent bay. Conventional hand electric and Unionmelt submerged arc welding facilities are installed, the latter with the necessary gantries and rotating gear for internal and external longitudinal and circumferential welds, and the former with the usual fixed floor plates and mechanical manipulators. Equipment includes heavy shears capable of cutting 1½-in. thick plate cold, a Sedgewick folder of 10 ft. × ¾ in. capacity, flanging and joggling presses, a large cold saw, profile burners and 10 ft. bending rolls. A further set of rolls 15 ft. 6 in. long × 1 in. thick is on order. An old 8,000 press has been modernised and is used for exceptional bending work, including the



Lomatic II—A combined machine for drawing, cutting,

manipulation of slabs up to 13 ft. 6 in. wide, 15 in. thick, and weighing up to 60 tons. Where necessary the Company is able to utilise certain equipment of John Brown and Co., Ltd., Clydebank, including rolls taking material up to 40 ft. in length and a vertical plate-bending press with a capacity of 3 in. thick cold, 5 in. thick hot, on plates up to 20 ft. wide.

The works is well situated to use economically a variety of the adjacent steelworks heat-treatment furnaces, able to accommodate the largest work, and also has under construction its own gas-fired stress relieving furnace of such capacity, adjustable in size, as to be able to take assemblies up to 52 ft. long, 20 ft. wide and 17 ft. high. Situated beside this furnace is a large Jackman shot blast chamber, 35 ft. long, 20 ft. wide and 17 ft. high.

For examination of weldments there are available a 300 kV Philips X-ray unit and a radium bomb for gamma-ray examination of greater thicknesses.

Machining

For the machining of the large castings, forgings and weldments which form part of these heavy engineering structures, there is available a comprehensive range of machine tools manufactured by Asquith, Cravens, Giddings and Lewis, Kendal and Gent, Kitchen and Wade, Noble and Lund, Richards, Swift, etc. They include planers up to 16 ft. 6 in. wide, 15 ft. high, 39 ft. stroke; plano millers up to 13 ft. square \times 26 ft. stroke; vertical boring mills up to 17 ft. 3 in. diameter; 10½ in. spindle horizontal borer; and a number of 8 in., 7 in. 6 in., and smaller floor and table type borers. A lathe swinging 6 ft. \times 40 ft. between centres is awaiting delivery to replace an older machine installed temporarily. A full use is made of portable tools, including a 3 in. Giddings and Lewis horizontal borer, a radial drill of 4 in. spindle, a number of smaller units and a push-stroke shaper.

Erection

For erection purposes, various portions of the floor have been provided with T-slotted beams and plates. Additionally a number of pits have been provided—one with a floor of adjustable depth, for medium presses;



Housing and working tables of a Loewy 40 in. blooming mill in course of erection.

one of "stepped" construction to accommodate the largest rolling mills; and a really impressive pit of exceptional size for the largest presses, giving 60 ft. clear headroom under the 75-ton cranes. It is provided with horizontal and vertical T-slotted beams to facilitate lining up; full services of electricity, hydraulic power, compressed air; an elevator from pit floor to crane level and points on which to erect tubular scaffolding.

The Roll Shop

An item of obvious importance in any rolling mill is the roll itself. Whether it shall be of cast iron, cast steel or forged steel, what surface hardness it should possess and other factors are largely based on experience and, to some extent on individual preference. Visitors were able to see the facilities available, in the Firth-Brown roll shop, for the production and heat-treatment of various types from cogging mill rolls to cold finishing rolls, ground and buffed to the beautiful mirror finish on which the quality of the finished sheet and strip depends.

Quality Control

As those with experience of large forgings, castings and weldments will appreciate, careful inspection and testing

FLYING SHEAR

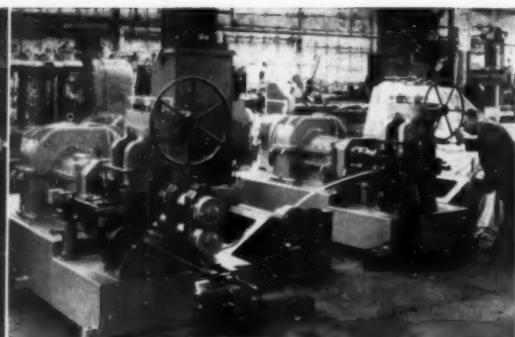
STRAIGHTENER AND POLISHER FOR ROUND STOCK



and straightening round, hexagon and square rods.



A ring rolling mill



A cold-strip mill

is necessary to ensure their soundness and serviceability. The Company is fortunate in having the experience and knowledge of its associated Companies at its disposal and, in particular, in having the facilities of the Brown-Firth Research Laboratories available for the solution of any chemical, physical and metallurgical problems which arise. Reference has already been made to radiographic facilities; these together with the more recently developed supersonic flaw detection equipment are available to supplement the more generally used methods of metallurgical inspection.

Light Machines, Ltd.

As mentioned above, this Company was established to manufacture specialised precision equipment for hydraulic machinery and prototypes of new equipment. Recently a new machine was built there for drawing, cutting-to-length, straightening and polishing extruded rods in one continuous operation at high speed. Hitherto, such rods, after being produced on a hydraulic extrusion press, have been drawn, cut-to-length and polished on separate machines involving much handling time and uneconomical use of skilled labour. The new machine is 82 ft. long and is a combined unit producing a large output of finished rods of precise dimensions and

requires the services of only one operator. It has now been installed in a well-known Midland Works and is already arousing considerable interest both at home and overseas.

Another machine recently completed for export by Light Machines, Ltd., is a ring rolling mill. This mill produces steel and other rings to finished dimensions, directly from pierced blanks without the more usual intermediate process calling for extensive plant equipment. The mill commences with a roughly pierced blank and manufactures rings in a range of sizes from 6-in. bore up to 50 in. outside diameter. There is a wide range of application for rings of this type including, for example, ball races, gearing rings and wheel treads, single and double flanged tyres used for overhead crane wheels, etc.

Other specialised plant seen by visitors at Yeovil included a metal powder press, a die-casting machine, a three-throw high-pressure pump, and a strip rolling mill for Sweden for rolling down extruded strips of non-ferrous metals for manufacturing zip-fasteners.

Acknowledgment is made to Thos. Firth and John Brown, Ltd., and to the Loewy Engineering Co., Ltd., for the photographs used to illustrate this article.

The Argon Arc Welding Process

In a recent paper, presented at the recent Autumn Conference of the S.A.S.M.U.T.A., on the above subject, Mr. W. K. B. Marshall, assistant director of the British Welding Research Association, emphasised that argon arc welding is a new process to industry and new applications are being found for it every day. In less than 10 years the process has developed from an interesting experimental effort in welding magnesium alloys to one which can now be applied to most of the common metals and alloys. Many metallurgical, electrical and practical problems have been solved during this period, particularly those relating to the welding of aluminium and its alloys. The main virtue of the process as far as aluminium is concerned is that welding can be carried out without the use of a flux and this enables fillet and lap welds to be used with confidence. The main problem which was encountered in the early experiments on aluminium was that due to the rectification effect when using the A.C. argon arc. This has now been overcome from the practical point of view although it still requires considerable investigation.

Because of the cost of the argon gas, the cost of welding aluminium and its alloys by the process is still high for

normal fabrication shops but the introduction of mechanical welding with higher rates of travel considerably reduce costs and in these circumstances the process may be competitive. The application of the process to heavier aluminium sections in this country awaits the development of heavier equipment than that which is at present available.

The use of argon arc welding for stainless steel is proceeding with very little trouble and the process now competes in certain ranges of thickness with metal arc welding. The welding of copper by the argon arc process, however, is still not satisfactory due mainly to the porosity encountered in the finished welds. Similar trouble arises when welding nickel and Monel, but Nimonic and Inconel appear to give satisfactory welds.

A new development of the process in America consists in replacing the tungsten electrode by aluminium wire which is fed forward rapidly. This makes the process less cumbersome and permits ready welding in the overhead and other difficult positions. Another development is that of of poke welding in which overlapping sheets are welded together by melting from one side. This process has considerable application where the equivalent of spot welding is required from one side of the work.

Some Physical Properties of Aluminium Alloys at Elevated Temperatures

Communication from the National Physical Laboratory

Results are given for the thermal conductivities, electrical resistivities and coefficients of linear thermal expansion of nine wrought and ten cast aluminium alloys, mainly over the range 20° to 300 °C., but in a few instances to higher temperatures. These alloys were intended for use in aero engines and some had been given special heat treatments at about 160° C. before being submitted for test. Heating above this temperature was observed to produce considerable changes in the properties of some of the alloys and in these cases values have been obtained both for the "as received" states and after sufficient heating at 300° C. for the changes to reach completion. With two of the alloys heating beyond 300° C. was observed to produce further changes in conductivity and these have been studied in greater detail.

It is shown that, apart from a few exceptions, mainly confined to the alloys which are rich in silicon, the conductivity results can all be represented to within about 5% by the equation

$$K = 0.53 \times 10^{-8} T \rho^{-1} + 0.025$$

where K is the thermal conductivity expressed in calories per square cm. per second for 1 cm. thickness and 1° C. difference in temperature, ρ is the electrical resistivity expressed in ohms. per square cm. per cm., and T is the absolute temperature. This equation provides a basis for estimating the thermal conductivities of other aluminium alloys.

With regard to the coefficients of expansion it is found that these decrease as a result of heat treatment and in some of the alloys this is accompanied by a considerable increase or decrease of the original length of the specimen. These changes do not appear to be related systematically with the corresponding changes in electrical resistivity.

Introduction

THE work described in the present paper formed part of an investigation into the properties of aluminium alloys undertaken at the National Physical Laboratory on specimens supplied by the Royal Aircraft Establishment. Part 1 deals with the thermal conductivity and electrical resistivity of these alloys, and Part 2 deals with their thermal expansion. The results of mechanical tests carried out in the Engineering Division of the Laboratory on many of the alloys are being published independently.¹

PART 1: THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY

By R. W. POWELL, D.Sc., Ph.D., F.Inst.P. and M. J. HICKMAN

Determinations of the thermal conductivities of various commercial and experimental aluminium alloys were made over the range of operational temperatures, to provide data which could be used in calculations relating to the flow of heat in the metallic parts of internal combustion engines. The measurements of electrical resistivity were to some extent incidental but were included for two reasons. Firstly, as a means for readily obtaining evidence for the effect of heat treatment on the thermal conductivity. Secondly, to provide additional information on the relationship between the thermal conductivity and electrical resistivity of the alloys tested. Previous investigations had shown that

these two properties can be correlated over a wide range of temperature for alloys of aluminium² copper³ iron⁴,⁵ and magnesium⁶, by plotting the thermal conductivity against the absolute temperature divided by the electrical resistivity. This aspect of the work is of practical value as well as of academic interest in that it provides a basis for the estimation of thermal conductivity from the very much easier measurement of electrical resistivity.⁷

Details of Alloys tested

Determinations of thermal conductivity and electrical resistivity have been made on nine wrought and ten cast aluminium alloys. Details regarding the chemical compositions and previous heat treatments were provided by the Royal Aircraft Establishment and are set out in Table I.

Experimental Determinations

The alloys were submitted in the form of bars from which test specimens measuring 10 in. in length and 1 in. in diameter could be machined. The number of bars of each alloy supplied ranged from two to six, and in order to ascertain their uniformity measurements were first made of the electrical resistivity of each rod at room temperature. In general the resistivities of the different bars of the same alloy agreed to within 2-3% and it was not considered necessary to make thermal conductivity measurements on all of the bars. One exception was the experimental R.A.E. alloy 55 for which the room temperature electrical resistivity values ranged from 5.5-6.1 microhm cm.²cm. for the wrought

¹ Thorpe, P. L., Tremain, G. R., and Ridley, R. W. In course of publication by Inst. of Metals.

² Kempf, L. W., Smith, C. S., and Taylor, C. S. *Amer. Inst. Min. and Metal Engrs. Trans.*, 124, Inst. Metals Divn., pp. 287-298 (1957).

³ Smith, C. S. and Palmer, E. W. *Amer. Inst. Min. and Metal Engrs. Trans.*, 117, p. 225 (1935).

⁴ Powell, R. W. Second Report of the Alloy Steels Research Committee, Iron and Steel Institute, Special Report No. 24, pp. 253-268, 1939.

⁵ Powell, R. W. and Hickman, M. J. Second Report of the Alloy Steels Research Committee, Iron and Steel Institute, Special Report No. 24, pp. 242-251, 1939.

⁶ Powell, R. W., *Phil. Mag.*, Ser. 7, 27, pp. 677-686, 1938.

⁷ Powell, R. W., *J. Iron and Steel Inst.*, 154, 116P-121P, 1946.

TABLE I.—CHEMICAL COMPOSITION AND HEAT TREATMENT OF ALUMINIUM ALLOYS.

WROUGHT ALLOYS:	B.S. Speci- fication	Bar Ref. No.	Chemical Composition, %											Heat Treatment	
			Cu	Ni	Mg	Fe	Si	Ti	Mn	Be	Cr	Zn	Co	Al	
RR 59	L42	—	2.31	1.20	1.46	1.23	0.88	0.07	—	—	—	—	—	—	Rest 2 hr. at 525° C., quenched; 16 hr. at 170° C., quenched.
Y alloy	3L25	—	3.76	1.85	1.33	0.40	0.45	—	—	—	—	—	—	—	511° C., quenched in fairly hot water; aged at room temp.
Lo-Er	D.T.D. 324	—	1.03	1.03	0.91	0.50	11.80	0.02	0.03	—	—	—	—	—	522° C. for 12 hr., quenched; aged 4 hr. at 135° C., cooled in air; aged 4 hr. at 200° C., air cooled.
RAE 40C	—	—	2.0	5.0	0.5	0.5	0.3	—	3.0	0.4	0.5	—	—	—	6 hr. at 570° ± 5° C., cold-water quenched; 20 hr. at 150° C., air cooled.
RAE 47D	—	—	1.0	4.0	0.5	0.5	0.3	—	3.0	0.4	—	—	—	—	6 hr. at 570° ± 5° C., cold-water quenched; 20 hr. at 160° C., air cooled.
RAE 55	—	29A	1.68	3.05	0.50	0.39	0.19	0.08	1.98	—	0.45	—	—	—	4 hr. solution heat treatment at 570° C., quenched in boiling water, 40 hr. ageing at 160° C., cooled in air.
"	—	33A	1.67	2.85	0.52	0.41	0.17	0.07	2.02	—	0.49	—	—	—	"
"	—	39A	1.68	3.01	0.49	0.40	0.15	0.03	1.41	—	0.17	—	—	—	"
RR 77	D.T.D. 363	—	2.20	—	2.54	0.31	0.26	trace	0.51	—	—	1.96	—	—	2 hr. solution heat treatment at 450° C., quenched in water at 70° C., 4 hr. ageing at 135° C., air cooled.
RAE SA1	—	—	5.0	—	0.6	0.5	11.0	0.05	—	—	—	—	0.2	—	3 hr. at 495°–500° C., cold-water quenched, 16 hr. ageing at 165° C., air cooled.
RAE SA44	—	—	5.0	—	0.5	0.5	11.0	0.1	0.4	—	—	—	0.3	—	"
CAST ALLOYS:															
RR 50	D.T.D. 133B	—	1.40	0.90	0.12	1.18	2.25	0.19	—	—	—	—	—	—	10 hr. at 160°–170° C., air cooled.
RR 53C	D.T.D. 309	—	1.33	0.87	0.50	1.12	2.42	0.15	—	—	—	—	—	—	2 hr. at 530° C., water quenched, 15 hr. at 160°–170° C.
RR 131D	—	—	0.20	1.20	1.39	0.30	0.50	0.12	0.44	—	0.18	0.45	0.25	—	10 hr. at 160°–170° C., air cooled.
Alpax Gamma	—	—	—	—	0.35	0.28	12.0	—	0.29	—	—	—	—	—	4 hr. at 510°–518° C., cold-water quenched, 16 hr. at 150°–165° C.
RAE 40C	—	—	2.0	5.0	0.5	0.5	0.3	—	3.0	0.4	0.5	—	—	—	6 hr. at 570° ± 5° C., cold-water quenched, 20 hr. at 150° C., air cooled.
RAE 47B	—	—	1.0	4.0	0.5	0.5	0.2	0.2	3.0	—	—	—	—	—	Tested in "sand cast" and "chill cast" conditions.
RAE 55 (chill cast) . . .	—	—	1.89	2.90	0.56	0.43	0.21	0.07	1.55	—	0.15	—	—	—	4 hr. solution heat treatment at 570° C., boiling water quenched, 12 hr. at 200° C., air cooled.
RAE SA1 (chill cast) . . .	—	—	5.0	—	0.6	0.5	11.0	0.05	—	—	—	—	0.2	—	3 hr. at 495°–500° C., cold-water quenched, 16 hr. ageing at 165° C., air cooled.
RAE SA44 (chill cast) . . .	—	—	5.0	—	0.5	0.5	11.0	0.1	0.4	—	—	—	0.3	—	"

condition and from 6.3–6.6 for the chill-cast condition. Subsequent chemical analysis of the wrought bars disclosed that with this alloy segregation had occurred, particularly of the manganese, chromium and titanium constituents (see Table I). In this instance the thermal conductivity measurements were made on bars of high and of low resistivity. The individual resistivities of bars of SA1 and SA44 also differed from the mean value by ± 5 to $\pm 8\%$. These bars contained numerous blow-holes in the chill-cast samples and some cracks in the forged samples.

For the purposes of the thermal conductivity determination each 1-in. diameter bar was drilled and tapped at one end and screwed to a standard bar of known thermal conductivity and of similar diameter. A heating coil was inserted in a cavity in the free end of the standard bar and the free end of the test bar was fitted with a cooling unit embodying a water-flow calorimeter. The composite bar was mounted centrally within a vertical guard tube with the standard bar uppermost and the interspace was packed with heat-insulating powder. A steady temperature distribution was maintained in the system and lateral heat leakage prevented by suitable adjustment of the temperature of the guard tube. The apparatus used is illustrated in Fig. 165 of an earlier paper.⁵ The temperature gradients in the test and standard bars were obtained from readings of thermocouples composed of 40 S.W.G. Nichrome and Constantan wires attached at points along their lengths, and the heat flow in the test specimen was derived both from the gradient set up in the standard bar and from a determination of the heat absorbed by the water-flow calorimeter. These two measurements were invariably in close agreement, after small corrections were applied

to allow for any imperfections in the match between specimen and guard tube.

The thermal conductivity, K , was then calculated from the equation—

$$K = QS/A (T_1 - T_2)$$

where Q is the mean of these two corrected values for the quantity of heat flowing in the bar in calories, A is the cross-sectional area of the bar in sq. cm. and T_1 and T_2 are the temperatures in °C. at two points on the rod separated by a distance of S cm.

Provision was also made for passing a direct current of the order of 10 amp. through the bars so as to enable the electrical resistivity of the alloy under test to be measured from time to time. Using the thermocouple wires as potential leads the electrical resistivity could be determined by comparing the potential drop across a length of the bar with that across a standard resistance connected in the same circuit and carrying the same current. All voltage readings were taken on a potentiometer reading to 1 micro-volt.

Tests were in general made at several temperatures up to a mean of 300° C. after which a repeat observation was made at a lower temperature. It soon became apparent that the heat treatment involved in the test was causing changes in the properties of some of the alloys. Y alloy and RR53C were two of the early alloys studied for which the repeat values obtained after heating to high temperatures indicated that the heat treatment had appreciably improved both the thermal and electrical conductivity. The nature of this change in the case of Y alloy is shown in Fig. 1.

In order to obtain information as to the temperature at which this change in conductivity had occurred determinations were made of the room temperature

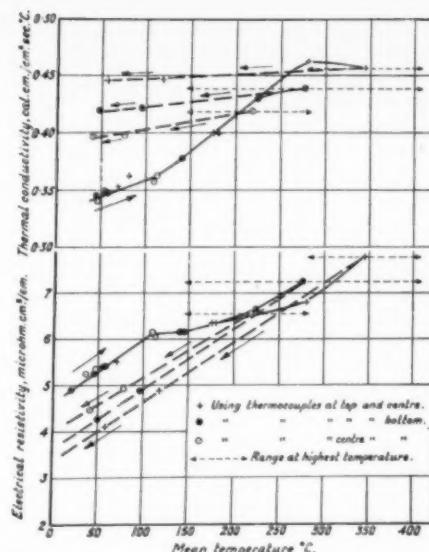


Fig. 1.—Y alloy : Thermal conductivity and electrical resistivity against mean temperature commencing in as received condition (quenched from 511° C. in hot water and aged at room temperature).

resistivity of various 4-cm. sections of the bars. From the observed temperature distribution along the bar when it had been heated to the maximum temperature it is possible to assign to each section the temperatures at which it had been heat treated. These results are set out in Table II for the bar of RR53C alloy.

TABLE II.—EFFECT OF HEAT TREATMENT AT VARIOUS TEMPERATURES ON THE ELECTRICAL RESISTIVITY OF ALLOY RR53C AT ROOM TEMPERATURE.
(Initial electrical resistivity = 5.1₂ microhm cm²/cm.)

Temperature of heat treatment—i.e., maximum temperatures reached by ends of section. (°C.)	Mean heat-treatment temperature for section. (°C.)	Electrical resistivity at room temperature. (microhm cm ² /cm.)
85—175	130	5.1 ₂
109—197	152	5.1 ₂
153—245	199	4.1 ₂
173—267	221	4.1 ₂
198—280	239	4.1 ₂
245—318	281	4.1 ₂
280—354	317	4.0 ₂

It will be seen that the decrease in resistivity set in after part of the bar had been heated above 175° C. This alloy had originally been aged at 160°—170° C. for 15 hours, so it appears that the change commences when the ageing temperature is exceeded.

In view of these changes it was decided that the investigation should be extended to include measurements made on specimens of each alloy after they had been uniformly heated at about 300° C. until the changes reached completion, and then air cooled.

Observations of the electrical resistivity at room temperature were made before and after this heat treatment, and the values obtained for each alloy are set out in Table III.

It will be seen that the heat treatment caused appreciable changes in the electrical resistivities of all the alloys, with the exception of RR 131D, and in six instances the changes produced ranged from 20–30%.

The value of 3.5 microhm. cm.²/cm. obtained for Y alloy is of interest in that it is less than the room tem-

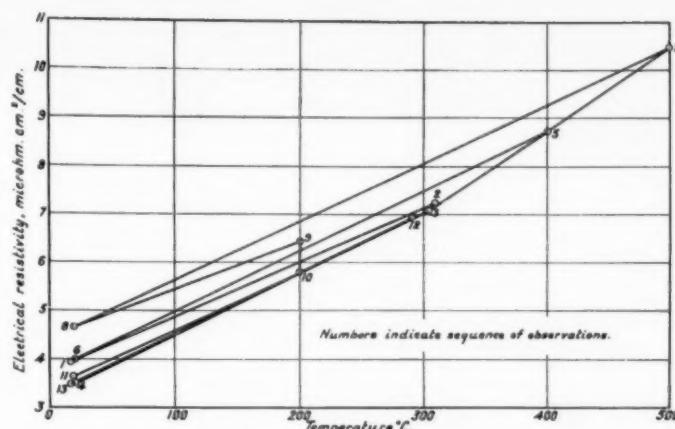


Fig. 2.—Y alloy : Electrical resistivity at various uniform temperatures for specimen which had received the treatment indicated in Fig. 1.

TABLE III.—EFFECT OF HEAT TREATMENT AT 300° C. ON THE ROOM TEMPERATURE ELECTRICAL RESISTIVITY.

Material	Temperature (°C.)	Electrical resistivity : (microhm cm ² /cm.)		Decrease in electrical resistivity : (%)
		Condition as received : see Table I	After heat treatment at 300° C.	
WROUGHT ALLOYS :				
RR 59	16	4.6 ₂	3.8 ₂	17
Y alloy	16	4.9	3.5	29
Lo-Ex	14	4.7	4.1 ₂	12
40C	20	6.0	5.0	17
47D	20	5.2	4.6	12
R.A.E. 55 (Bar 39A)	20	5.5 ₂	5.0	10
R.A.E. 55 (Bar 33A)	20	6.1	5.4 ₂	11
RR 77	20	5.5	5.2	5 ₂
SA 44	20	5.6	4.3	23
SA 1	20	5.7	4.1	28
CAST ALLOYS :				
RR 50	16	4.9	4.3	12
RR 53C	16	5.1	4.1	20
RR 131D	16	6.5	6.4	1 ₂
Alpax Gamma	16	4.4	3.8 ₂	12 ₂
40C	14	8.1	7.6 ₂	5 ₂
47B	20	9.5	9.0	5 ₂
47B (chill cast)	20	9.3	8.7	6 ₂
R.A.E. 55 (chill cast)	20	6.3	5.9	6 ₂
SA 44 (chill cast)	20	7.0	5.2	26
SA 1 (chill cast)	20	6.4 ₂	4.9	24

perature values obtained for the three heat-treated sections of the bar of this alloy for which results were given in Fig. 1 and which had been heated to a little above 400° C. at one end. This indication that further changes had occurred on heating above 300° C. was confirmed by a more detailed study of the dependence of the electrical resistivity of Y alloy on heat treatment.

The specimen on which the results given in Fig. 1 had been obtained was mounted in a furnace in which it could be uniformly heated, and the sequence of electrical resistivity determinations shown in Fig. 2 were obtained. The resistivity was measured across the same section of the bar as before, so the initial room temperature resistivity value, marked 1 in Fig. 2, agrees with that shown in Fig. 1 by the line through the ringed crosses. The effect of heat treatment on the electrical resistivity can be seen by following the numbered observations. At each elevated temperature sufficient time was allowed for approximate equilibrium to be reached, then the bar was removed from the furnace and allowed to air-cool before taking the room-temperature measurement. It will be seen that after being held at 300° C. the room-temperature resistivity had fallen to the anticipated value of 3.5 microhm. cm.²/cm. Heating at 400° C.

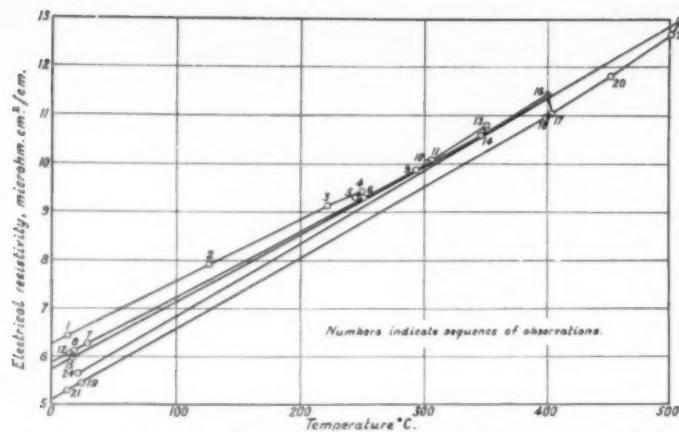


Fig. 3. Chill cast RAE 55 alloy : Variation of electrical resistivity with temperature.

TABLE IV.—THE ELECTRICAL RESISTIVITY AND THERMAL CONDUCTIVITY OF WROUGHT ALUMINIUM ALLOYS

Material	Condition AR = as received (see Table I) H = Heat treated at 300° C. and air cooled.	Temperature (°C.)	Electrical resistivity (microhm. cm./cm.)	Thermal conductivity (cal. cm./cm. sec. °C.)	Condition AR = as received (see Table I) H = Heat treated at 300° C. H400 = Heat treated at 400° C.	Material	Temperature (°C.)	Electrical resistivity (microhm. cm./cm.)	Thermal conductivity (cal. cm./cm. sec. °C.)
Y alloy	AR	20	4.7	0.55 ⁰	RR 53C	AR	20	4.9 _b	0.34 ⁰
	H	20	3.8	(0.40 _b)		H	20	4.3 _b	(0.38 _b)
		100	1.9	0.42			100	5.5	0.40
		200	6.2 _b	0.45 _b			200	6.9	0.41
		300	7.6	0.44			300	—	(0.42)
		20	4.9 _b	(0.35 _b)			20	5.2 _b	(0.32)
Lo.Eu	AR	20	5.5 _b	0.36	RR 131 D	AR	20	6.3 _b	0.34
	H	20	6.3 _b	0.41 _b		H	20	7.4	0.37 ⁰
		100	3.5	(0.43 _b)			100	4.2	(0.39 _b)
		200	1.5	0.45			200	5.3	0.40 ⁰
		300	5.7	0.46 _b			200	6.7	0.41 _b
		20	4.8	(0.38 _b)			300	8.0 _b	0.41 _b
40 C	AR	20	6.1	0.38 _b	40C, Sand cast	AR	20	6.6 _b	(0.25 _b)
	H	20	7.4	0.40 _b		H	20	7.5 _b	0.28 _b
		100	4.2	(0.41 _b)			100	8.8	0.31 _b
		200	1.2 _b	(0.41 _b)			200	9.7 _b	0.34 _b
		300	5.5 _b	0.42 _b			20	0.27 _b	
		20	7.0	0.42 _b			20	4.4	0.39 _b
17 D	AR	20	5.2	0.32 ⁰	Alpsax	AR	20	5.8	(0.45 _b)
	H	20	4.6	(0.31 _b)		H	20	5.0	0.45
		100	5.7	0.36 _b			100	6.4	0.44 _b
		200	7.0	0.37 _b			200	7.8 _b	0.44
		300	8.3	0.38 _b			200	8.2	(0.21)
		20	5.5	(0.30 _b)			100	9.5	0.23 _b
RR 77	AR	20	6.2	0.34 _b	17B, Sand cast	AR	20	7.8	(0.22 _b)
	H	20	4.2	(0.39 _b)		H	20	9.0	0.24
		100	5.2	0.41 _b			100	10.6	0.26
		200	6.1 _b	0.42 _b			200	12.4	0.27 _b
		300	7.8	0.42 _b			200	9.5	0.19 ⁰
		20	6.1	(0.28 _b)			200	13.3	0.19 ⁰
R.A.E. alloy 55 (Bar No. 33A)	AR	20	7.1	0.31 _b	R.A.E. alloy 55 _b , Chill cast	AR	20	6.6	(0.26 _b)
	H	20	8.2	0.34 _b		H	20	7.6 _b	0.28 _b
		100	5.2 _b	(0.31 _b)			100	8.9	0.31
		200	6.5	0.33 _b			200	9.9 _b	(0.28 _b)
		300	7.8	0.35 _b			200	7.1	0.30 _b
		20	9.2	0.37 _b			200	8.4 _b	0.33 _b
R.A.E. alloy 55 (Bar No. 39A)	AR	20	5.5 _b	0.31 ⁰	17B, Chill cast	AR	20	9.0	0.34 _b
	H	20	5.0	(0.34 _b)		H	20	5.2	(0.31 _b)
		100	6.0	0.36 _b			100	6.4	0.33 _b
		200	7.2	0.38 _b			200	7.8	0.34 _b
		300	8.4	0.39 _b			200	9.3	0.36
		20	5.8	(0.31 _b)			300	10.0	0.38
SA 1	AR	20	5.8	0.31 _b	SA 1, Chill cast	AR	20	6.3	(0.33 _b)
	H	20	6.9	0.33 _b		H	20	7.4	0.38
		100	1.1	(0.43 _b)			100	4.9	(0.39 _b)
		200	5.3	0.42 _b			100	6.3	0.39
		300	6.8	0.42 _b			200	8.2	0.39
		20	8.2	0.42 _b			200	10.0	0.38
SA 44	AR	20	5.5	(0.32 _b)	SA 44, Chill cast	AR	20	7.0	(0.30 _b)
	H	20	6.6	0.34 _b		H	20	8.5	0.32
		100	1.3	(0.39 _b)			100	5.2	(0.29 _b)
		200	5.4	0.40 _b			100	6.7	0.37
		300	8.4	0.41 _b			200	8.7	0.37
		20	8.4	0.41 _b			300	10.5	0.37

caused a definite increase in resistivity. Following the heat treatment at 500° C. the bar was water-quenched to see if its resistivity returned to the original as received value. It was lower, but was increasing with time. The full room-temperature ageing change had not reached completion before the specimen was introduced into the furnace at 200° C. At this temperature a steady decrease occurred, and after five days the subsequent room-temperature value had fallen to 3.65 mierohm.cm.²/cm. After taking the bar up to 290° C. and cooling a value of 3.5 was once again obtained.

This experiment shows the extent to which the resistivity of Y alloy is dependent on its thermal history, and confirms that heat

TABLE V.—THE ELECTRICAL RESISTIVITY AND THERMAL CONDUCTIVITY OF CAST ALUMINUM ALLOYS.

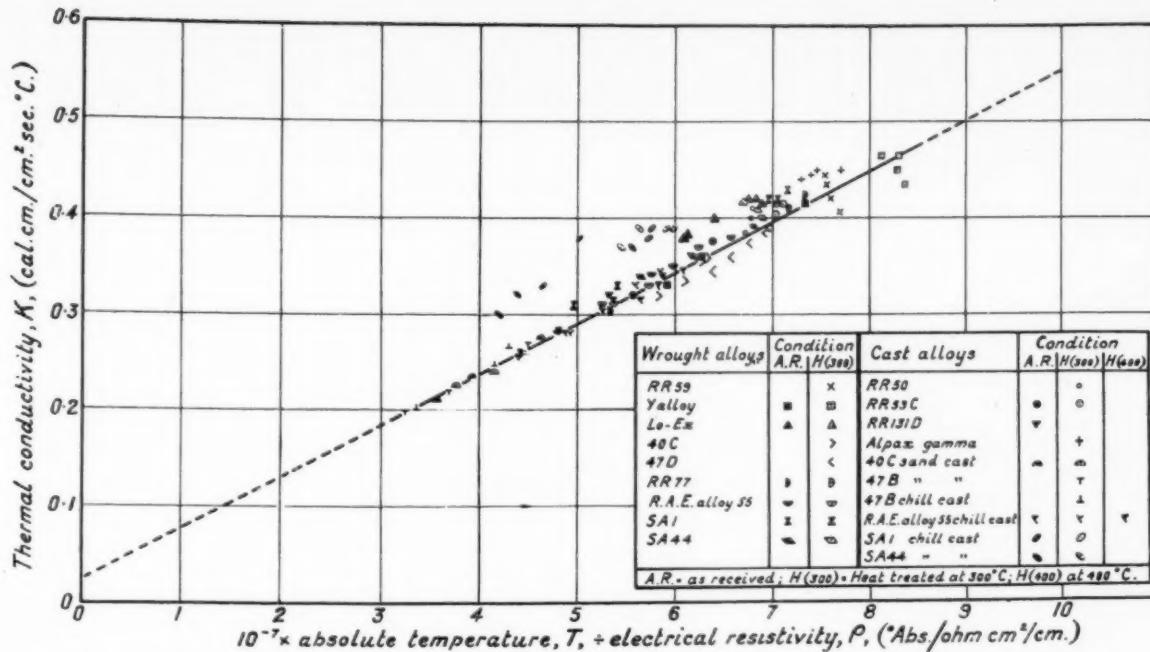


Fig. 4. -Wrought and cast aluminium alloys ; thermal conductivity against absolute temperature divided by electrical resistivity.

treatment above 300°C. produces changes in the opposite direction to those produced in the range of 200°-300°C.

Similar tests were later made on a bar of the chill-cast RAE 55 alloy, commencing in the as-received condition. With this material a further decrease in room-temperature resistivity occurred after heating to 400°C. These results are plotted in Fig. 3. Since this alloy had been aged for 12 hours at 200°C. before being sent for test no further ageing tests were made at this temperature, but the bar was aged for periods of about 20 hours at intervals of approximately 50°C. from 250°-500°C. and cooled to room temperature after each 50°C. step. From Fig. 3 it will be seen that a definite decrease in resistivity occurred at about 250°C. and that the corresponding reduction in resistivity at room temperature is from 6.5 to 6.1 microhm. $\text{cm.}^2/\text{cm.}$ This is of the same order as the reduction previously noted in Table III for another bar of this alloy after heating to 300°C. No further decrease is obtained on heating the present bar to 300°C., indicating that the change in the alloy which is responsible for this part of the observed effect appears to be capable of reaching completion at 250°C. On heating to higher temperatures there are indications of another change taking place in the alloy, a small decrease in resistivity being observed at 350°C. and a larger one at 400°C. After being heated to 400°C. the resistivity at room temperature is 5.3₅ microhm. $\text{cm.}^2/\text{cm.}$ There is no further change after heating to 450°C., but after 500°C. a definite increase is seen to have occurred.

The foregoing results indicate that the electrical resistivity of RAE 55 alloy is also very dependent on its thermal history. The same was found to be true of its thermal conductivity. For this alloy thermal conductivity determinations have been made over the temperature range 40°-300°C. for one bar commencing in the as-received condition and for another bar after the normal

heat treatment at 300°C. and also after subsequent ageing at 400°C.

Tables IV and V contain values for the thermal conductivities and electrical resistivities of all the alloys tested. These values have been read off at temperatures of 20°, 100°, 200° and 300°C. from smooth curves drawn through each set of experimental points. To obtain the thermal conductivity at 20°C. a small extrapolation has been necessary, since the lowest mean temperature at which values were obtained was usually about 40°C.

The present results have all been plotted in Fig. 4 in an analogous manner to that suggested by Kempf, Smith and Taylor.² Apart from the alloys of high silicon content, which Kempf, *et al.* also found to behave abnormally, these results are seen to conform fairly well to the straight line.

$$K = 0.53 \times 10^{-8} T \rho^{-1} + 0.025 \dots \dots \dots (1)$$

where K is the thermal conductivity expressed in calories per sq. cm. per sec. for 1 cm. thickness and 1°C. difference in temperature, T is the absolute temperature and ρ the electrical resistivity in ohms. $\text{sq. cm.}/\text{cm.}$ This line is displaced about 5% above that suggested by Kempf, Smith and Taylor.

It will be seen that the agreement is similar for the as-received and heat-treated states and tends to improve with increase in temperature. The results for only three of the alloys depart from the line by more than 8%. These are the wrought Lo-Ex alloy and the cast alloys SA.1 and SA.44 all of which contain large amounts of silicon and give points displaced above the line. The greater departures observed with the two cast alloys may be associated with their porosity.

It appears from these results that equation (1) can be used for the estimation of the thermal conductivity of an aluminium alloy from a knowledge of its electrical resistivity. In most instances this estimated value should not differ from the true thermal conductivity by

more than about 5%, but with silicon-rich alloys the estimated value is likely to be low. Due allowance can be made for this when the composition of the alloy is known. The values marked by asterisks in Table IV and V have been estimated in this way.

PART 2: LINEAR THERMAL EXPANSION

By C. R. BARBER, B.Sc.

Concurrently with the investigation of the electrical resistivities and thermal conductivities described in Part 1 measurements were made of the linear thermal expansion coefficients of specimens of the same alloy and an account of this section of the work is given here.

The method of measurement employed was that in which the relative expansion of a specimen and its silica container is measured by a dial gauge (see Fig. 5). This method has been in use for many years, but the details of the mode of operation of the apparatus and its particular form may be of interest in showing the precautions taken to obtain reliable results.

The specimen used is cylindrical in shape, 1.4 cm. in diameter and 30 cm. in length, with its ends finished plane and perpendicular to the axis. It is contained in a silica tube of internal diameter 1.7 cm. and 60 cm. in length, which is closed at one end, this closed end being re-entrant to give a hemispherical surface on which the specimen rests. A distance piece of silica in the form of a cylindrical rod or tube of about the same diameter as the specimen, is hemispherical at its lower end and rests on the plane upper surface of the specimen. By this means the difference of expansion between the test-piece and the silica containing tube is transmitted to a dial gauge attached rigidly to the top of the tube. The upper end of the distance piece of silica is ground plane and perpendicular to its axis and the ball-end of the plunger of the gauge rests on this surface. Both the specimen and distance piece are bound in two places on each by fine asbestos twine to obtain a good sliding fit in the containing tube. A steel collar is cemented to the top of the silica tube and this is gripped by a steel chuck having attached to it a steel-ring in which the gauge is firmly clamped. By this arrangement it is not necessary to make accurate adjustments of lengths of the containing tube, specimen and distance pieces as the chuck can be moved on the steel collar until the required gauge reading is obtained and then clamped in this position. The apparatus, thus assembled, may be moved about and even shaken without alteration of the gauge reading. The gauge usually employed has a sensitivity of 0.01 mm. per division.

The standard procedure adopted for determining the expansion is first to immerse the tube in a bath of melting ice to a depth of 45 cm. and obtain the length at 0°C. Sufficient time is given, of course, for the test piece to attain the temperature of the bath; usually about 1 hour is necessary, since the specimen is somewhat large. The apparatus is then removed from the ice, the tube dried and immersed to the same depth in a bath of well-stirred oil maintained at the desired temperature until a steady gauge reading is given. The ice value is redetermined to check that no permanent change in the length of the specimen has occurred. The maximum temperature obtainable in the oil bath is 300°C. Higher temperatures, up to 600°C., can be obtained by the use of a stirred molten salt bath. These stirred-liquid baths are those in regular use for the

calibration of thermocouples and resistance thermometers. It is considered that the temperature uniformity of the specimen under these conditions is one or two tenths of a degree centigrade and that the temperature of the specimen is known to this order of accuracy. The inaccuracy in the determination on account of temperature measurement error is thus negligible. By use of a comparatively long test-piece, moreover, a substantial expansion is obtained and the limitation on accuracy of the measurement is the reliability of the dial gauge. The gauges employed are examined and calibrated in the Metrology Division of the Laboratory.

The procedure above-described was adopted in the first determinations of the expansions of the aluminium alloys, that is to say, the cycle of heating was 0°, 100°, 0°, 200°, 0°, 300°, 0°C. It was found, however, that some specimens did not reproduce the zero reading, showing a marked growth after exposure to the high temperature, and it was realised that the alloys as received were not necessarily in a fully stabilised condition and the test procedure was accordingly modified to avoid the alternations of high and low temperature consequent upon intermediate zero observations. The revised procedure was as follows:—A zero observation was first made and then the apparatus was placed in the oil bath and the temperature slowly raised to 300°C., the temperature rise being checked at 100°, 200° and 300°C. for a sufficient time to obtain steady gauge readings. The specimen was then allowed to cool slowly in the bath and a final reading at 0°C. was taken. Under these conditions it was possible in some cases to obtain reproducible zero readings on specimens in the "as-received" condition. The alloys which changed in length by an appreciable amount after expansion measurements up to 300°C. are set out in Table VII showing the percentage change which occurred. It will be observed that alloys Alpax Gamma, RR. 50, RR. 53 and SA. 1 all showed increases of length of about 0.05%. On the other hand alloy RR. 77 decreased in length by about the same amount. It is for this reason that it has not been possible to record coefficients for these alloys in the "as-received" condition, for there was a time-temperature change of the effective zero taking place in the course of the observations. In others the coefficient for the range 0°–100°C. only has been given since it was established that no appreciable change took

Fig. 5.—Expansion apparatus.

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TABLE VI.—PERCENTAGE CHANGE IN LENGTH OF SPECIMENS AT 0°C. AFTER HEATING TO 300°C.

Alpax Gamma	RR 50	RR 53C	Lo-Ex	RR 77	SA 1		SA 41	
					Cast	Cast	Forged	Forged
+0.04	+0.05	+0.05	+0.03	-0.06	+0.06	+0.07	+0.02	+0.01

place in this temperature range in the course of the experiment.

In view of the above-described changes it was decided with many of the alloys to give a stability heat treatment at 300° C. and to make further measurements with the specimen in this condition.

The mean coefficients of linear thermal expansion for all the alloys tested are given in Table VIII for the temperature ranges 20°–100° C., 20°–200° C. and 20°–300° C. In calculating the coefficients a correction has been applied for the coefficient of expansion of silica which has been taken to be 0.5×10^{-6} per 1° C. The measurements were all made with reference to a zero of 0° C. and the expansion relative to 20° C. deduced therefrom. It will be noted that the expansion of the heat-treated specimen is always lower than that of the "as received," with the exception of Y alloy which showed a very small increase in coefficient after heat treatment, but this is not significant, particularly as there was a 10% variation in the coefficient among the several specimens examined. It is of interest to note in contrast that, as a result of heat treatment, the electrical resistivity of this alloy shows the largest change of any (see Table III, Part 1). A general comparison of the results do not show any obvious correlation between the changes in resistivity and expansion coefficient.

Acknowledgments

Miss N. Beer, B.Sc., rendered assistance in connection with the conductivity tests on the alloys SA. 1 and SA. 44. Mr. L. H. Pemberton carried out some of the earlier measurements of thermal expansion.

Staff Changes and Appointments

MR. W. E. BARDGETT, of the Central Research Department, United Steel Companies Ltd., has succeeded Dr. J. E. Hurst as representative of the Institution of Metallurgists on the Council of the Iron and Steel Institute.

MR. R. HOWARD, A.M.I.Mech.E. has been appointed Chief Technical Engineer of the Railway Division of British Timkin Ltd.

MR. OLIVER W. HAYES is now associated with the National Research Corporation, Cambridge, Mass., where he is the Corporation's Patent Counsel.

MR. V. C. FAULKNER has been elected an honorary life member of the Institute of British Foundrymen, in recognition of the important contributions that he has made to the progress of the Institute and of the foundry industry.

MR. G. D. ELLIOTT, Ironworks Manager at the Appleby Frodingham plant is visiting the U.S.A. to address a special joint meeting of the Chicago and Eastern States Districts of the Blast Furnace and Coke Oven Association on the subject of "Carbon as a Blast Furnace Refractory."

LORD HALSBURY, PROFESSOR R. S. EDWARDS, MR. A. H. WILSON and MR. A. H. S. HINCHCLIFFE have been appointed Members of the Advisory Council for Scientific and Industrial Research for five years, as from October 1st, 1949. They succeed Mr. W. F. Lutgens, Mr. W. J. Drummond, Sir Henry Guy and Sir William Halcrow, who have retired on completion of their appointments.

TABLE VII.—LINEAR THERMAL EXPANSION OF WROUGHT AND CAST ALUMINIUM ALLOYS.

Alloys	Condition AR = as received H = Heat treated	Mean coefficient of linear expansion per 1° C. $\times 10^6$		
		20°–100° C.	20°–200° C.	20°–300° C.
WROUGHT:				
RR 59	H	21.4	22.3	23.6
" Y" (3L25)	AR	21.4	22.5	23.8
" Y" (3L25)	H	21.5	22.7	23.6
" Lo Ex"	H	19.4	20.6	21.5
40C	AR	20.3	21.5	22.5
40C	H	20.1	21.2	22.0
47D	AR	21.3	22.0	22.6
47D	H	20.9	21.7	22.6
SA 1	AR	19.6	—	—
SA 1	H	19.4	21.1	21.6
SA 44	AR	19.1	—	—
SA 44	H	19.0	20.3	20.6
RR 77	AR	23.6	—	—
R.A.E. 55 (29A)	H	22.9	23.6	25.4
R.A.E. (39A)	AR	21.9	22.3	23.0
CAST:				
RR 50	H	22.1	22.9	23.6
RR 53D	H	24.5	24.6	24.4
RR 131D	H	23.8	23.2	22.6
Alpax Gamma	AR	20.0	—	—
Alpax Gamma	H	20.0	21.0	21.8
40C	AR	21.0	22.0	22.7
40C	H	20.8	21.9	22.7
47B	AR	21.9	22.4	23.4
47D (chill cast)	AR	21.0	21.7	22.6
SA 1 (chill cast)	AR	20.1	—	—
SA 1 (chill cast)	H	19.9	20.9	21.4
SA 44 (chill cast)	AR	19.7	—	—
SA 44	H	19.7	20.6	21.3
R.A.E. 55	AR	22.2	22.8	23.8

The work described above was carried out in the Physics Division of the National Physical Laboratory on behalf of the Ministry of Supply. This paper is published by permission of the Director of the Laboratory with the approval of the Ministry of Supply.

MR. A. E. FRANKS has joined the Metals Research Department of the National Research Corporation, Cambridge, Mass., where he will be engaged in the development of new products and of high vacuum furnaces for metallurgical processing.

The Iron and Steel Institute Hatfield Memorial Lecture

THE fourth Hatfield Memorial Lecture was delivered by Sir Andrew McCance, D.Sc., LL.D., F.R.S., on Friday, October 28th, in the Firth Hall of the University of Sheffield. The Vice-Chancellor of the University, Dr. J. I. O. Masson, LL.D., D.Sc., F.R.S., was in the Chair.

In his introductory remarks, Sir Andrew said he welcomed the opportunity of paying tribute to the work of Dr. Hatfield, whose friendship he enjoyed for many years. That the occasion should take place in his native town of Sheffield, where his life's work was carried out, and to the development of whose special steel industry he made many important contributions, and that it should take place within the precincts of the University where he received his early training, seemed specially appropriate. After referring to the catholicity of Dr. Hatfield's interest in metallurgy, Sir Andrew said he had chosen as his subject "The Plastic Behaviour of Solids" because it was one of Hatfield's interests and because it was a subject to which he himself had given much thought.

At the conclusion of the lecture, thanks were expressed by Professor H. W. Swift of the Engineering Department of the University.

Italian Research on Electro-Deposition of Metals

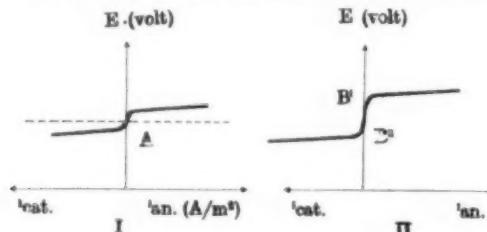
The Work of Professor R. Piontelli

Considerable work on the electrodeposition of metals and alloys has been carried out by Prof. Piontelli, much of which is not readily available in English, and this summary of some of his work will be of interest. The field covered comprises the general electro-chemical behaviour of metals, their behaviour in concentration cells, electrodeposition of alloys, and auto-displacement of lead.

FOR many years the researches undertaken at Milan University in the Electrochemical Dept., by Prof. Roberto Piontelli and his co-workers have been of great interest and increasing importance, both in the practical and theoretical sphere. Some of the results have been published in papers in the Italian technical and scientific press, notably during the last year or two (e.g., *Gazz. Chim. Ital. et al.*); and one or two were included in the Proc. of the 3rd Internat. Electrodep. Conference, London, 1947. Apart from these latter however, much of the work does not appear to be readily available in English, and it is therefore proposed in this article to attempt a brief summary from a selection of Prof. Piontelli's recent papers issued last year. No claim to anything like completeness can be made; and all the papers will doubtless appear in due course in the abstract journals.

The field to be covered herein comprises the general electro-chemical behaviour of metals, their behaviour in concentration cells, electro-deposition of alloys, and auto-displacement of lead.

In a contribution to *La Ricerca Scientifica* 1948, 18 (7), 824-830, Piontelli discusses some of the theoretical aspects of electro-chemical behaviour. Metals may be divided into (a) quasi-ideal or normal, such as Pb, Tl, Hg, Cd, etc.; (b) inert metals (in transition to incomplete electronic substrata); and (c) intermediate. Some of the features relevant to such classification are indicated, including chiefly electro-motive behaviour, polarisation, barrier effects, which are closely related to hydrogen action and physical properties of materials involved. Metal classes (a) and (b) may be distinguished graphically by the two curves herewith I and II, where A (in I), no current flowing, corresponds to thermodynamic data; and when current is flowing changes are very small. But in II the difference is more marked:



From the physico-chemical nature of the inert metals in the metallic and ionic (solution) phase, it may probably be inferred that when ions pass into solution "formation" and "separation" of these ions should

be completed before there can be any real effect of hydration groups or centres (campi di idratazione); and, on the other hand, in the separation of the hydrated ions, dehydration should take place before there can be effective action of metallic ion groups (campi di legame metallico). In both cases, there may be barrier effects to be superadded to electrical action. It is concluded provisionally that kinetic behaviour depends essentially on barrier effects, but this part of the work, more thoroughly treated, forms the subject of a paper still in the press (a month or two ago). Reference is made to L. Pauling (*Phys. Rev.* 1938, 54, 899; *J. Am. Chem. Soc.*, 1947, 69, 542) in connection with the chemical and physical points of view for electro-chemically inert metals; also to papers by von Hevesy and others, for some confirmation of these views.

The author proceeds to consider the relation between this inertia and anodic passivity with particular reference to the work of U. R. Evans, W. J. Müller, etc., and the effect of covering films (M. Pourbaix, *Journées des Etats de Surface*, Paris, 1945, p. 251). But a purely thermodynamic-structural interpretation leaves unexplained certain features, as follows: If the kinetic point of view is essential in studying ionic exchange, and inertia due to barrier effects be assumed, then the wide variation in passivity manifestations shown in different metals may be at least partly explained as due to: oxidation effects in relation to nature of film formed, and other changes on the metal surface, such as OH⁻ ion absorption due in turn to conditions of c.d. and other conditions, which may sometimes lead to porous film formation, with varying ohmic resistance and polarisation.

A special difficulty in the study of passivity is the anion effect, e.g., on deformation, electronic affinity, free ion radius, etc. This effect is discussed both for normal and for inert metals; together with the depassivating action of the halogens. The effect of halogen ions on deformation, conductivity, etc. is tabulated. From the foregoing considerations it is hoped that some gaps in the passivation theory may be filled.

In the first of a series of notes on the cathodic and anodic behaviour of metals (*Gazz. Chim. Ital.*, 1948, 78, 717-24) Piontelli and Poli deal with some of the difficulties encountered in measuring anodic and cathodic polarisation, especially that directly concerned with ion exchanges at the metal/solution interface, thus involving barrier effects; and describe a new method based on (a) use of electrodes prepared under standardised conditions, preferably by electro-deposition; (b) use of cylindrical cells with electrodes occupying the entire cross section, thus securing uniformity of current

distribution ; (c) direct determination of local potentials, e.g., with the Luggin capillary ; (d) vigorous stirring near the electrode, for example, by bubbling through nitrogen completely free from oxygen ; and (e) measuring polarisation from a lengthy series of c.d. values, each of which is kept constant till the corresponding potential is stabilised.

Apparatus used is shown in Fig. 1. It consists of a glass tube C to which are attached tubes G and G¹ serving respectively as inlet and outlet for the gas bubbled through. Electrodes E and E¹ are attached through wires H and H¹ to rubber stoppers T and T¹ which close ends of tube C without coming into appreciable contact with the solution which, in this particular set-up has no action on the stoppers. Methods used when this arrangement is not possible are described in subsequent notes. The reference electrode R is immersed in tube F fixed in stopper T and reduced down to capillary size to pass through a small hole in electrode E. The electrodes were of electrolytic Pb (99.995%), and several lead salts were used in solutions, e.g., the sulphamate, nitrate, fluoroborate, perchlorate, etc. Instruments used included an Allochio-Bacchini potentiometer, Multiflex-Lange galvanometer, and a Weston "Universal" for c.d. Temps. were 25°, 50° and 75° C. Results are tabulated and the general conclusions are :—

- (1) Lead is typically normal in the sense defined, distinguished by anodic and cathodic barrier effects, somewhat limited, according with its behaviour in concentration cells and in deposition and auto-displacement reactions (see below).
- (2) Barrier effects are appreciably affected by the nature of the anions. In the type of reference here used the effect of solution used on equilibrium potentials was eliminated. The equilibrium potential values with respect to the calomel electrode indicated that there was no evident relation between ion activity and barrier effects.
- (3) Relation between anions and barrier effects is largely analogous in anodic and cathodic processes. Thus, among the different metals, those which show high barrier effects with current in one direction show them also with opposite current.
- (4) Barrier effects are particularly small with sulphamate and nitrate solutions ; somewhat larger with methyl-sulphate ; and larger still with fluoroboric, perchloric, acetic and sulphanilic solutions.
- (5) Generally, except in the case of acetate solutions—discussed in a subsequent note—the most marked barrier effects are met in solutions forming deposits of compact (dense) micro-crystalline structure.
- (6) Increase in temp. tends to depress barrier effects.

Auto-displacement of Lead

This forms the subject of two notes (*Gazz. Chim. It.*, 1948, **78**, 599–606, 606–613). The principal type actions taking place when a metal, in contact with a solution of its salt, undergoes ionic exchange therewith, are indicated, according as the metal and/or solution is homogeneous or heterogeneous. The example of lead in certain solutions—nitric or nitro-acetic—is discussed in connection with a brief literature review (Senderens, Heller, Cohen and Heldermaann, Thiel, and H. S. Rawdon) etc. The main object of the present work was a study of the formation of new individual products or crystalline

aggregates clearly distinguishable at the surface of test metal ; without however neglecting to note phenomena such as the formation of nitro-nitrite, which may throw some light on mechanism.

In studying the effect of the nature of solution the following factors were considered : (a) *kind of anion*, which played a decisive role in individual crystal formation only in solutions (active) containing NO_3^- ions or some nitro derivatives. Solutions used included acetic, perchloric, etc., which separately or without additives showed little action. But those with nitric

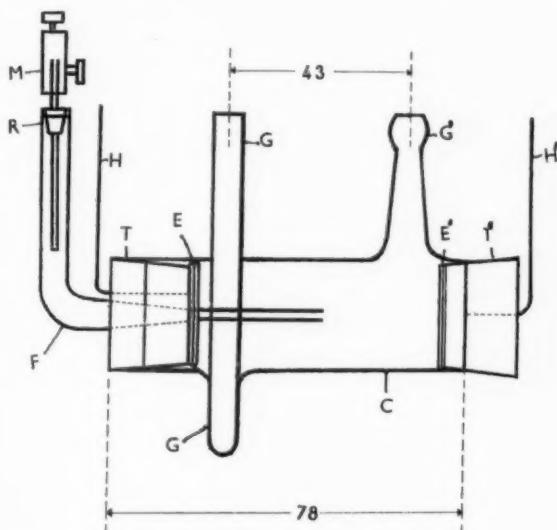


Fig. 1

anions, or containing additive such as sodium nitrite, or mixed lead salts, were much more interesting ; and still more so with solutions of nitromethane with lead sulphamate or acetate wherein there was very rapid formation of new crystals parallel with marked metallographic action. Organic solution mixtures favourable or otherwise to dendritic formations are specially noted. (b) *Lead ion concentration*.—Extent and rate of visible auto-displacement increases with Pb ion concn. shown in tests with the nitrate solutions of 0.01–0.5 m/lit. (c) *Free acidity*.—This, in the solutions just mentioned, reduced formation of dendrites. (d) *Oxidising agents*.—Addition of potass. permanganate (10g/lit.) to a lead nitrate soln., although a layer of MnO_2 was formed, did not definitely affect dendritic formation. (e) *Colloids, inhibitors, and misc.*.—Gelatine proved slightly depressive ; thio-semicarbazide was wholly or nearly inhibitive ; thio-urea (8g/lit.) was completely inhibitive ; and diphenyl-thiourea somewhat less so. (f) *Dissolved oxygen*.—As the solutions were thoroughly de-aerated this effect could not be properly ascertained, though the complete elimination of oxygen seemed to retard somewhat dendritic formation. (g) *Copper salts* appeared to accelerate attack on test sample and eliminate formation of Pb dendrites.

In the second note other factors are considered ; surface treatment, preparation and purity of sample ; and some further observations made on conditions and nature of crystal formation and distribution especially the dendritic forms.

From tests with other metals it appears that lead is the only one that clearly exhibits auto-displacement, with visible formation of new crystalline structure. Even the other metals which, like lead, are electro-chemically normal : Sn, Cd, Tl, Zn, etc., and the intermediates, Ag, Bi, do not show such displacement, except possibly through formation of concentration cells and especially in bimetallic systems. Tests with the nitrates of Bi and Tl were negative in regard to dendritic formation. Various questions arising from this singular behaviour of lead are considered at some length. Such behaviour is certainly consistent with the normal character of lead and absence of barrier polarisation effects ; also with certain structural properties of this metal, such as low cohesion energy, tendency to recrystallise even at low temp., and easy auto-diffusion, together with low ionic hydration.

The question of free energy also appears to be of special interest and is considered in connection with the appearance of new centres (germi) of crystallisation and anion effects. The present experiments have shown that the presence in soln. of the NO_3^- ion, or of nitromethane or the like, is decisive ; although, on the other hand, with mixed solutions of nitric and other ions the action of the NO_3^- is reduced. Some data are presented from unpublished work of Piontelli and Poli on anodic and cathodic polarisation values for lead in solutions containing diverse anions.

The general conclusions are : Study of dendritic formation in auto-displacement of lead, in solutions containing nitric or nitromethane (or some other nitro derivative) ions, shows that such formation depends on several factors, but is only slightly affected by purity of the lead and its particular form—re-melted, laminated, electro-deposited. And it is not to be explained in the way hitherto proposed, but appears to be due essentially to the particular kind of ionic exchange taking place with lead, usually very active ; under conditions where local causes favour separation of ions and formation of new activity centres (germi) not obstructed by those already well developed. Among these conditions the nature of the anion occupies a prominent place.

Behaviour of Metals in Concentration Cells

This subject is dealt with in four notes (*Gazz. Chim. Ital.*, 1948, **78**, 424-9, 430-5, 614-622, 697-702). The principal objects were to study the nature of ions present and more especially to study the operation of concentration cells of the type : $\text{Me}_1/\text{NaX}_{\text{aq}}//\text{Me}_2\text{X}_{\text{aq}}/\text{Me}_3$, in which anode and cathode are the same metal Me_1 , but different from that (Me_2) in solution. This is of particular interest when Me_1 belongs to the inert class and is not much less noble than Me_2 which is of the normal type.

In the first series of tests of the extended programme (*loc. cit.* 614-22) the test metals were tin, and iron in chloride solutions (HCl, LiCl, BaCl₂, MgCl₂).^{*} There was no H₂ evolution with tin, but quite definitely with iron ; and in each case there was cathodic formation of crystals and some anode attack, the latter being less with Fe than with Sn. In the 2nd series, with lead, cadmium, iron, copper, nickel, varying combinations of solutions (upper and lower) were used consisting of (1) sodium and lead nitrates ; (2) lead sulphamate and

sulphamic acid ; (3) sodium and cadmium chlorides ; (4) sodium and copper sulphates or chlorides.

Results again confirmed the normal behaviour of Pb, Sn, Cd, Cu. These cells were compared with those in which electrodes of same dimensions but of inert metals were used. In these latter there was not only more free energy available but cathodic polarisation effects were also eliminated, i.e., one cause of low functioning in concentration cells using inert metals only. With cells of inert metal anode the nature of the anion may be of the greatest importance compared with the known diversity in anionic activating power. On the other hand, with solutions of the HCl type (activated) with addition of various chlorides (of Li, Ba, Mg) the effect, most marked with Sn, is much less with Fe.

Further tests were made, qualitatively with the test metal placed vertically, and quantitatively with the two electrodes placed horizontally ; the metals being bismuth, antimony, and arsenic. Solutions upper and lower consisted of HCl (3.33N) and the respective chloride in HCl solution. Test metals were the three named, and also Fe or Ni. Bi proved to be normal, whilst there was a tendency to inertia in antimony which was still more marked with arsenic. There was marked diversity, too, in the structure of the electro-deposits or displacements. Whilst Bi forms in a state of extreme sub-division it yet appears definitely crystalline ; but antimony is amorphous (the so-called explosive form) ; as also is arsenic. The different form of inertia in this latter from that of other metals of the class will be considered in a subsequent note.

Since writing the foregoing the fourth note of the series dealing more in detail with the work with Bi, Sb, and As, has been received from Prof. Piontelli (*loc. cit.*, pp. 697-702) ; and it is again confirmed that, whilst bismuth is normal, with little or no inertia, antimony definitely shows inertia, and this is still more the case with arsenic. There are differences also in the form or nature of the deposition ; bismuth, although in very finely divided state, is nevertheless definitely crystalline ; antimony, on the other hand, is amorphous, and arsenic quite definitely so.

The third note of the series, also lately received, sums up the results of work with Sn, Pb, Cd, and Cu, in concentration cell tests. Comparison has been made between these cells and those formed by substituting for both electrodes others of same dimension but of electro-chemically inert metal (Fe, Ni), and of nobility not much below that of the metal in solution. Fe took the place of Pb and Cd, and Ni replaced Cu. In these substituted cells there was not only greater free energy but cathodic polarisation was also eliminated—the principal cause of low efficiency in inert metal cells. In this latter type of cell the nature of the anion becomes very important in respect to the varying "activating power" of anions, as exemplified in the experiments with iron electrodes in solutions containing NH_4SO_3 and NO_3^- anions.

Electro-deposition of Alloys

This forms the subject also of four notes : the first two in *La Metallurgia Ital.*, 1942, **34** ; the third in *Proc. 3rd Internat. Electrodep. Conf.*, London, 1947 pp. 121-125 ; and the fourth in *La Met. Ital.* 1948, **40**, 3-8. Special attention is given in Nos. 1, 2 and 4 to theory, for it is rightly held that without an adequate theoretical basis the study of the many complex

* These formed the upper solution ; the lower consisted of chlorides of the metal under test.

phenomena of electro-deposition is liable to "shipwreck on the rocks of empiricism." The present summary is taken mainly from Note 4.

Earlier considerations had shown that deposition solns. of salts of two or more metals depends largely on the following: (a) relative nobility of metal under static conditions—absence of current; (b) sum of irreversible actions accompanying deposition of each metal; (c) nature and course of current movements, diffusion and convection, on which real concentration of the various metals at the cathode depends; and their variation with time.

In each c.c. of solution near an electrode, the rate of separation of ions contained therein, referred to a cm^2 of electrode surface expressed as c.d. (cathodic i_c), may be shown as: $i_c = Dn e^{-U}$ (1) wherein Dn is a frequency factor indicating number of ions striking 1 cm^2 of surface per sec. D is therefore proportional to \sqrt{T} , and $-U$ indicates the probability with which each ion may be expected to attain one of the energy states whereby its separation is possible; γ represents mean probability of effective separation. An analogous expression is found in which $-Q$ replaces $-U$ to indicate the probability of a constituent having sufficient energy to pass into solution and δ is the mean probability, thus $i_a = A \delta e^{-Q}$ (2) representing the velocity with which metallic phase constituent passes into solution in ionic form. U and Q may be potential differences at contact between metal and solution.

Polarisation curves may be represented by

$$\Delta E_A = A + B \ln i \quad \dots \dots \dots \quad (3)$$

from which the following very possible relations derive:

$$U = U'_o + \sigma' z \epsilon E_A + \sigma'' z \epsilon \Delta E_A$$

$$Q = Q'_o - \tau' z \epsilon E_A - \tau'' z \epsilon \Delta E_A \quad \dots \dots \dots \quad (4)$$

wherein U'_o and Q'_o are functions of absolute temp. T and of other physico-chemical factors excluding potential difference, z is ion valency, ϵ is electron charge, and σ' , σ'' , τ' , τ'' are proportionality coeffs.

The next two expressions (5) and (6) derived from above represent 1° , E_A , and ΔE_A under static (non-current) conditions. From these and Nernst's equation for RT , values of current density i are given (7, 8, 9, 10, 11) when the electrode is subjected to a polarisation: $\Delta E_A < 0$ and under other conditions. An expression is also given for metal nobility grade. The general formula (10) shows $i = f(n_o, A, I, H, I, \dots)$ where A is additional current carrying capacity, I total cathodic

$\frac{D_o z F}{d}$ current, H is $\frac{1}{d}$ and U is mobility; the expression thus indicates dependence of deposition current on conditions of electrolysis. In the case of alloy deposition

$$i = H(n_o - n) - \frac{\gamma n_o U I}{A - \gamma n_o U} \quad \text{In this case some of}$$

the factors involved will depend on concentration etc. of additional ions, such e.g. as those of CN in cyanide solutions.

There is not space to follow the mathematical treatment further for alloy deposition and that from simple or complex salts. It is emphasised that a study of the characteristic structure of alloy deposits cannot be wholly based on considerations such as those which have been briefly noted and which can serve merely as a guide. A typical example is the alloy Pb-Sn which forms dense crystalline deposits, even in conditions which, for the separate metals, would give macro-

crystalline deposits without much change in rate of deposition.

The structure of a deposit depends, generally, on the relative velocity of: (1) separation of metal deposited in positions of growth of incomplete reticulated planes; (2) formation of new two-dimensional centres (germi); (3) formation of centres or groups (germi) of new crystalline individuals. With normal metals all these processes develop without barrier effects; but the simultaneous deposition of two of these metals may lead to considerable change in structural character without any energy effects of importance; even when, owing to their immiscibility, mixed stable phases are not attained, and regular growth of centres or groups (germi) is prevented, as with lead-tin alloys. Decrease in size of crystal grains in alloy deposition, compared with that of single metals, appears to be a matter of general experience.

Nature of the phases forming depends on: firstly, factors governing formation of mixed phases from solution mixtures, e.g., atomic and electronic conditions and relations, more or less electronegative state, etc.; secondly, conditions of deposition (Cf. M. Volmer on *Kinetik der Phasenbildung*, Dresden, 1939, p. 200). Application of the phase rule, so far as primary formation of the less stable phases is concerned, has been frequently questioned. In any case, its validity in electro-deposition of alloys appears to be in doubt. The probability therefore of formation and growth of deposition centres (germi) is not directly related to stability of the corresponding phases. Such probability indeed is governed by factors such as: interfacial energy, barrier effects, and others which, in turn, are related, indirectly at least, to the thermodynamic stability of the final phase.

It is therefore, the more likely that, owing to disturbing causes, e.g., local variations in concn., adsorption, hydrogen occlusion, presence of anions and additives, phases may form which are not provided for in equilibrium diagrams of mixed liquid systems. Our principal aim must be to ascertain the existence, nature, and formation conditions of the mixed phases, and compare these with those indicated, from similar average mixtures, by the equilibrium diagram.

The further notes in this series on other alloys besides Pb-Sn and Ni-Co , namely Cu-Sn , Cu-Pb , and Cu-Au , and X-ray studies of same, are not yet available for the purpose of this survey.

At the London Conference* in 1947 (*loc. cit.*), however, R. Piontelli and L. Canonica, described deposition of Ni-Co , Pb-Sn , and Cd-Zn alloys in sulphamic or amido-sulphonic salt baths ($\text{NH}_2\text{SO}_3\text{H}$). And at the same Conference Piontelli presented a paper on effect of the basis metal on electro-deposition.

* Organised by the Electro-depositors Tech. Soc., 27, Islington High Street, London, N.I., by whom the Proceedings have now been published.

A Horizontal Surface Grinder for Railway Axle Housings

A twin head Plano type horizontal surface grinder, claimed to be the largest yet built, was recently landed in this country from the United States after being on order for over eighteen months. The machine is approximately 35 ft. long, 16 ft. wide and 12 ft. high. It has been designed and built by the Thompson Grinder Company of Springfield, Ohio, to the specification of British Timken Limited for use on railway axle housing production at their Birmingham works.

Effect of Homogenising on the Structure and Properties of an Extruded Al-Cu-Mg Alloy

By H. A. Unckel

The transverse tensile properties of extruded aluminium alloys of the Duralumin type are lowered by the presence of strings of inclusions of intermetallic compounds. The author has carried out an investigation into the effect of a homogenising treatment before and after extrusion and the results are presented in this article. In a subsequent article he will deal with the effect of such treatments on the fatigue properties.

EXTRUDED sections of commercial aluminium-copper-magnesium alloys usually contain two kinds of structural heterogeneity. The first consists of segregation of the ternary eutectic at the grain boundaries. The last portion of the melt to solidify contains copper and magnesium in the eutectic proportion and is thus considerably richer in these components than the bulk of the material. The time taken to heat the cast billet to the temperature of extrusion is generally insufficient to allow the eutectic to pass into solid solution. The second type of heterogeneity consists of inclusions of various intermetallic compounds of the alloying elements and impurities, with each other and with aluminium. Besides copper and magnesium, manganese is deliberately added to the extent of about 0.5% in order to give improved properties to the alloy. Iron and silicon in amounts of some tenths of a per cent. are always found as impurities in commercial alloys. Since only very small quantities of these elements are retained in solid solution, "intermediate constituents" or intermetallic compounds may be formed, or, as in the case of silicon, the component may occur in elementary form in the structure.

By the extrusion process the inhomogeneities or inclusions, as we may call them, are stretched into fibres or streaks parallel to the axis of the rod, and, since most of the intermetallic compounds are brittle, they are broken up and the fragments arrange themselves in a streak-like form. The presence of the inclusions explains the properties of the material, especially with specimens from the transverse direction where the applied stress

lies perpendicularly to the direction of the streaks. This is particularly true for parts machined from extruded stock. Previous experiments with rods of 50 mm. diam. have shown that the difference in properties between the longitudinal and transverse directions is especially pronounced with alloys of the Al-Cu-Mg. type¹.

Method of Investigation

In the present experiments extruded rods of greater diameter, namely 100 mm. were used, and it was to be expected, therefore, that the inclusions would not be excessively broken up and distributed by the extrusion process. One billet, 175 mm. diam. and about 1,500 mm. long was divided into three parts, each 450 mm. long, the remainder being reserved for specimens in the unextruded condition. Analysis of the material showed: 4.6% Cu, 1.35% Mg, 0.59% Mn, 0.25% Si, 0.42% Fe. The three billets were treated differently: One was heated to the extrusion temperature of 490°C. in about 3 hours, and was then immediately extruded. The second billet was held at 490°C. for 5 hours after this temperature had been attained, and the third was held for 15 hours at the extrusion temperature. For the heating, a temperature-controlled air-circulation furnace was used, the diameter of the container being 180 mm. Each billet was extruded to a diameter of 100 mm.

From the front end of the extruded rods a length of about 150 mm. was discarded, since the front end of extruded bars is scarcely worked at all and consists chiefly of the as-cast structure. Specimens were taken

1 H. Unckel, Properties of extruded rods. *Metallwirtschaft*, 21, 531-538.

TABLE I. MECHANICAL PROPERTIES OF SPECIMENS FROM VARIOUS POSITIONS IN THE EXTRUDED ROD (AFTER QUENCHING AND AGEING).

Billet not Homogenised before extrusion.										Billet Homogenised 5 hours before extrusion						Billet Homogenised 15 hours before extrusion					
Material Homogenised after extrusion for										Material Homogenised after extrusion for						Material Homogenised after extrusion for					
	0 hrs.		5 hrs.		15 hrs.			0 hrs.		5 hrs.		15 hrs.			0 hrs.		5 hrs.		15 hrs.		
	Long	Trans.	Long	Trans.	Long	Trans.		Long	Trans.	Long	Trans.	Long	Trans.		Long	Trans.	Long	Trans.	Long	Trans.	
F. Y. U. E.	34.3	30.8	35.4	31.5	36.1	32.6	35.1	30.4	36.8	32.8	36.8	32.0	35.2	28.9	36.5	30.2	36.8	31.6			
	17.2	37.0	31.0	40.6	51.1C	39.4	50.8	39.5	52.3	41.2	50.9C	40.0	51.2	37.2	52.1	39.7	51.4C	39.7			
	11	4	15	5	15	6	13	5	16	5	13	7	13	6	15	5	13	7			
F. Y. U. E.	35.2	—	—	—	35.5	—	35.0	—	—	—	35.9	—	35.0	—	—	—	—	36.3	—		
	51.2P	—	—	—	50.3P	—	50.4P	—	—	—	51.5P	—	51.8P	—	—	—	—	51.0P	—		
R. Y. U. E.	35.8	30.5	36.6	31.3	37.6	31.1	36.1	30.4	36.9	31.7	37.8	30.7	36.0	30.5	38.4	31.1	36.5	30.6			
	51.3	39.6	51.9	39.2	53.5C	37.4	51.7	40.3	54.0	41.4	54.1C	39.5	52.8	40.7	54.1	40.9	54.0C	39.4			
	14	6	14	5	13	4	14	6	15	6	13	7	14	6	14	5	14	6			
R. Y. U. E.	37.9	—	—	—	37.1	—	35.4	—	—	—	37.8	—	36.9	—	—	—	—	36.6	—		
	53.2P	—	—	—	52.3P	—	53.0P	—	—	—	53.0P	—	53.2P	—	—	—	—	53.1P	—		
F = Front end of rod.		P = Specimen from periphery.		Y = Yield stress at 0.2% permanent elongation Kg./mm. ²		E = Elongation % (gauge length 40 mm.).		U = Tensile strength. Kg./mm. ²													

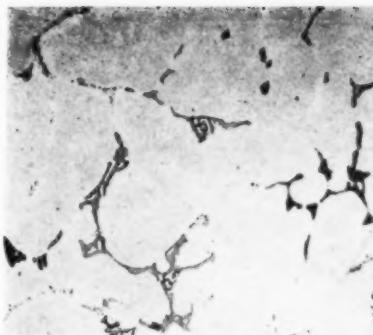


Fig. 1.—As-cast structure. $\times 150$.



Fig. 2.—Longitudinal section of extruded rod. Front end. Not homogenised before or after extrusion. $\times 150$.



Fig. 3.—Longitudinal section of extruded rod. Front end. Homogenised 15 hours before, and 15 hours after extrusion. $\times 150$.

in the longitudinal as well as in the transverse direction from the front and rear end of the bar (except the extreme front end), the longitudinal specimens being taken from both the periphery and from the centre of the section. For this purpose the rods were sawn into prismatical pieces $2 \times 2 \times 10$ cm. in the respective positions. A number of such pieces were also taken from the remainder of the cast billet. One group of specimens was heated to 490°C . for solution treatment and, on reaching this temperature, immediately quenched in water at room temperature. Solution treatment was carried out in a laboratory air-circulation furnace, the temperature being held constant by an automatic temperature controller actuated by a thermocouple which was inserted amongst the specimens. Heating took about an hour. A second group was held at 490°C . for a period of 5 hours, and a third for 15 hours at that temperature before water quenching. In this manner 9 different treatments were covered with zero, five, and fifteen hours homogenising before and after extrusion. Ageing was carried out at room temperature for one week and test specimens for tensile and fatigue testing were then machined from the prismatical pieces.

Test Results

The results of the tensile tests are collected in Tables I and II, each figure representing the mean of at least four specimens. It is noteworthy that the time of homogenising does not appreciably affect the mechanical

properties in excess of the usual scatter in the values obtained. The tensile strength varied between 50 and 53 kg./mm^2 for the longitudinal, and between 35 and 40 kg./mm^2 for the transverse specimens. The elongation was 10–15% (gauge length 40 mm.) in the longitudinal, and only 3–6% in the transverse direction, regardless of the duration of the homogenising treatment within the limits investigated. The yield stress was $35\text{--}38\text{ kg./mm}^2$ in the longitudinal and $28\text{--}32\text{ kg./mm}^2$ in the transverse direction. Thus, transverse specimens have a yield stress about 15% lower, an ultimate strength about 20% lower, and an elongation about 70% lower than longitudinal specimens.

It was found, further, that the position of the specimen with respect to the front or rear end of the bar and with respect to the position in the cross section (periphery or centre for the longitudinal specimens) had no marked influence.

TABLE II.—PROPERTIES IN THE UNEXTRUDED CONDITION AFTER VARIOUS HOMOGENISING TREATMENTS (QUENCHING AND AGEING)
(Mean of two values each).

	Not Homogenised		Homogenised 5 hrs.		Homogenised 15 hrs.	
	Long	Trans.	Long	Trans.	Long	Trans.
Y.	28.6	—	30.4	—	30.6	30.8
U.	29.7	29.3	32.6	28.9	34.5	32.1
R.	3	3	3	3	3	2

The Brinell hardness values varied from 105 for the cast material to 110–120 for the extruded material, all after quenching and ageing. Homogenisation was without effect on the hardness number.

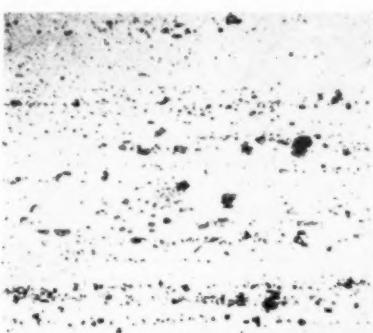


Fig. 4.—Longitudinal section of extruded rod. Rear end. Homogenised 15 hours after extrusion. $\times 150$.

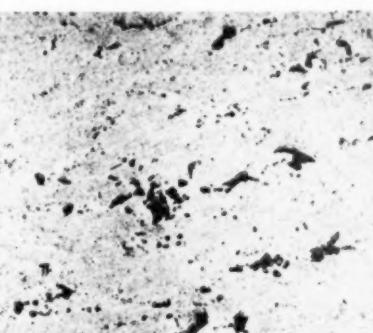


Fig. 5.—Transverse section of extruded rod. Front end. Homogenised 15 hours after extrusion. $\times 150$.



Fig. 6.—Longitudinal section of extruded rod. Front end. Homogenised 15 hours before extrusion and one week after extrusion. 490°C . $\times 150$.

The mechanical properties in the as-cast (not extruded) and heat-treated condition are, of course, considerably lower. The tensile strength was found to lie between 29 and 33 kg./mm.² and there was no evident difference between specimens from the axial and radial directions in the cast bar. The elongation was 2-4% and the yield point 28-30 kg./mm.². It is remarkable that the yield stress is only about 20% less than with the extruded and heat-treated material whereas the tensile strength is as much as 40% less.

Microscopic Examination

In the as-cast structure the ordinary intermediate phases are observed, namely, the binary and ternary eutectic, an iron-containing phase and a manganese-rich phase, all of which occur preferentially at the grain boundaries (Fig. 1).

By the extrusion process all these inclusions are stretched out to fibres in the direction of the bar, giving the appearance of a wood-fibre structure, especially in broken sections (Figs. 2-4).

In the transverse section the inclusions are randomly and more evenly distributed. (Fig. 5). In the longitudinal section the streaks are finer and narrower the nearer the rear end the specimen is taken, and the inclusions are broken into small particles. The time of homogenising had no perceptible influence on the amount of inclusions, after 15 hours homogenising, however, it could frequently be observed that the initially sharp-edged fragments had adopted a more rounded shape, clearly an effect of surface tension.

The fact that the streaks are finer towards the rear end of the bar is a consequence of the flow phenomena in the extrusion process. The material constituting the rear end of the bar has undergone a more intense deformation before leaving the container, as is well known. In the extreme front end of the rod (discarded for this investigation) the as-cast structure is almost preserved.

It is worth mentioning that homogenising periods up to one week did not dissolve the inclusions, as Fig. 6 shows, by comparison with the corresponding sample in Fig. 2, the amount of intermediate phase being about the same in both. Since intermetallic compounds in general are brittle even at the temperature of extrusion, they are broken up and therewith give rise to minute cracks between the fragments.

The fracture of the transverse specimens has a fibrous appearance as will be seen from Fig. 7, the fracture running perpendicularly to the axis of the specimen. With the longitudinal specimens the fracture has an irregular appearance or is inclined at an angle to the axis. Moreover, with the transverse specimens fracture mostly occurred at the same distance from the head of the specimen, at about one-fourth of the length of the sample, which was especially true for the specimens from the rear end of the bar. The almost invariable location of the fracture indicates that a weak zone



Fig. 7.—Fractures of tensile test pieces. Upper row. Not homogenised, lower row: homogenised 15 hours. The four nearest to the left: transverse specimens. The four nearest to the right: longitudinal specimens.

exists in the cross section of the extruded rod. As is easily understood from the mechanism of the extrusion process^{2,3} this zone coincides with the zone of maximum relative movement within the material in the neighbourhood of the orifice in the container, in which zone the inclusions are fragmented and fissures arise.

Discussion

There seems to be little doubt that the difference in properties in the longitudinal and transverse directions is caused by the streak-like arrangement of the inclusions. It could be argued, however, that a preferred crystallographic orientation of the individual crystal grains can play a part. As is known, for metals with a cubic face-centred lattice, to which class aluminium belongs, a preferred orientation is created by extrusion, the [111]—crystallographic direction lying parallel to the axis of the bar. Now, since the [111]—direction is the strongest one among the different crystallographic directions, a 10-15% higher strength can be expected in the longitudinal direction on account of the orientation effect. This applies, however, only if the reduction from billet diameter to rod diameter is considerable. With the present experiments the diameter of the rod was large in proportion to the diameter of the container or the billet, the reduction by extrusion being only about 65% so that any preferred orientation is incompletely developed. It is, therefore, most probable that the fibre-like arrangement of the brittle inclusions furnishes a full explanation of the effects observed. In the longitudinal direction the streaks act as strengtheners because they render yielding in this direction more difficult than if the material were homogeneous, whereas in the transverse direction, the streaks, and minute fissures, extended in the axial direction, lower the tensile strength and the ductility very considerably. In the tensile test, fracture occurs, so to speak, "prematurely" before the strain-hardening effect through plastic deformation during testing comes into play. This explains, too, why

2 C. E. Pearson, "The Extrusion of Metals," London, 1941.

3 H. Unckel, "Über die Fließbewegung, etc.," Berlin, Springer, 1928.

The difference in tensile strength between longitudinal and transverse specimens is much more pronounced than the difference in yield point.

It must be emphasised that in this investigation continuously cast water cooled billets have been used; for billets cast by other methods the results might be different. In the latter case, owing to the coarser structure, long-time homogenising might be of advantage

in order to re-dissolve precipitated phases. Furthermore, the overall values for strength and elongation in both longitudinal and transverse directions will continually decrease as the reduction by extrusion is further decreased, the values finally approaching those obtained in the as-cast condition.

The author's thanks are due to Mr. S. N. Karlsson for carrying out the tensile tests and to Mr. K. E. Jusselius for assistance with the metallographic work.

Aluminium in the Building Industry

REFERENCES have frequently been made in this Journal to the contribution the aluminium industry has made to the solution of the housing problem in the design and production of "temporary" bungalows. So rapid have been developments resulting from the construction of these earlier aluminium dwellings that aluminium buildings now being erected are regarded as permanent structures. The progress so far made has been such as to establish a new industry in this country which is not only solving structural problems at home but is substantially assisting the country's economic problems by successful overseas trading.

Some indication of the progress the aluminium industry has made in this field will be seen at the building exhibition to be held at Olympia, London, from November 17th to December 1st, 1949, inclusive. Here will be seen many developments which have resulted from the initial work on temporary bungalows, examples of which will be school buildings based on prefabricated unit construction and permanent aluminium bungalows for use in the tropics, where, in addition to ease of transportation, resistance to attack by insects and dimensional stability, the structure does not rot, warp or swell.

A thriving industry is already functioning and an interesting feature at the exhibition is a bungalow built partly of "Alframe" panels and partly of "Acrete" panels. The former consists of external corrugated aluminium alloy sheet, glass wool lining and fibre board interior. It is used for transportable housing, the largest contract to date by Structural and Mechanical Development Engineers, Ltd., who are displaying this bungalow, being for the Argentine Government—a complete "town" of 200 buildings was dispatched to that country last month.

The Acrete system of construction for permanent housing consists of aluminium framework filled in on site with local materials; an outstanding recent example of the use of this form of construction being the housing scheme at present being carried out in India. A survey of housing conditions in India was carried out by Mr. A. F. Hare, A.R.I.B.A., and, in conjunction with the engineering firm noted above, he produced a design for a semi-detached bungalow. The firm is creating a series of factories in India which will produce the foamed cement panels used in conjunction with the aluminium



Courtesy of Almin, Ltd.

Fig. 1.—Showing an "Alframe" aluminium alloy roof construction used in conjunction with pre-stressed concrete columns in a factory 800 ft. long and 60 ft. wide; by Structural and Mechanical Development Engineers, Ltd.

framework for these bungalows. The sections for the framework are produced by Southern Forge, Ltd., of Langley, and sent to India ready for assembling.

The application of aluminium alloy construction to factory buildings has also made considerable headway, notably in roof construction and for special structures. A special development has been its use in conjunction with pre-stressed pre-cast concrete columns, the most recent being the factory at Newport, Monmouthshire, for South Wales Board Mills, Ltd., shown above.

A PUBLICATION recently issued by Johnson, Matthey and Co., Ltd., 73/83, Hatton Gardens, E.C.1., deals with their Mallory products for resistance welding. The booklet is designed to be a work of reference for the welding engineer and its approach to the subject is practical throughout.

After a brief survey of the Mallory range of electrode and die materials it covers in turn the four main resistance welding processes—spot, seam, projection and flash and butt welding—in terms of the most suitable choice of material, the design and construction of the electrodes and recommended practice for obtaining maximum efficiency from machine material and electrode.

Corrosion and Corrosion Fatigue of Steel

Continued from page 10

There were possibilities of further acceleration of the tests by using greater speeds and higher temperatures. These were being investigated. The position was now that so many paints with good adhesion had been developed that either more intensive test conditions, or more sensitive methods of detecting paint failure were required. He thought that an electrochemical technique of measurement had shown promise and a combination of this with the rotor test might lead to further advances.

In discussion DR. HUDSON suggested that intensification of test conditions could be achieved by using a thinner paint film. In these tests hitherto the thickness of film tested had been up to 120 microns. In future the B.I.S.R.A. Corrosion Laboratory hoped to explore the possibility of reducing this thickness considerably.

The only comment he had on the C.R.L. apparatus was that the size of the specimen was limited to 1 in. sq. For certain purposes, particularly for tests on painting schemes, it was advantageous to use a large specimen. It was then easier to produce a surface that bore a closer relation to the surface condition over which paint was applied to ships' hulls in practice. B.I.S.R.A. were therefore developing a testing machine to take larger pieces; this would supplement rather than rival the C.R.L. apparatus.

DR. T. A. BANFIELD pointed out that if a thinner paint film were used, a shorter drying time would be necessary. DR. MAYNE raised the question as to whether the increased life of the paint coats observed on rusty steel was due to the chemical or physical nature of the surface.

In reply to the discussion DR. WORMWELL agreed that the thinner film would be worth trying, though it would have to be carefully controlled for uniformity.

Electrode Potential Measurement on Painted Steel

DR. WORMWELL also presented the last paper of the conference which was "Electrochemical studies of protective coating on metals," by F. WORMWELL, and D. M. BRASHER, Part I being on the above subject. This investigation arose naturally out of the studies of accelerated breakdown of paints. There was a recognised need for improved methods of assessing quantitatively the period of initial breakdown of paint systems on metals and also the extent of failure after a prescribed exposure period. The first technique selected was the measurement of electrode potentials of painted specimens immersed in artificial sea-water. By continuing the observations over longer periods than had been used by earlier workers, the electrical measurements could be closely correlated with the rate of breakdown of the paint as estimated visually and also by determinations of weight loss of the underlying metal. The technique had been used to investigate the effect of paint thickness, the type of surface finish, the method of treatment before painting, and the type of paint. These investigations were being followed up by conductance and capacitance measurements.

DR. HOAR thought potential-time curves gave interesting information and looked forward to their supplementation by the promised paper on conductance and capacitance of paint films. He pointed out that the curves were similar, though with a much longer time

scale, to those found with unpainted steel carrying only an oxide-film, and thought that they might be similarly explained.

He thought the technique valuable for exploring the mechanism of breakdown rather than for testing coatings. MR. BRITTON agreed with this view. The need for an objective test was emphasised by several speakers. Among them DR. MAYNE and MR. BATESON pointed out that all the four paints tested had a linseed oil vehicle, and wanted to know whether other media gave similar results.

MISS BRASHER in the course of her reply said that it was, indeed, important to use a good vehicle. With it good results could be obtained over a wide range of pigmentation.

Iron and Steel Institute Autumn Meeting

THE Autumn Meeting of the Iron and Steel Institute was held on November 10th and 11th at the Offices of the Institute, 4, Grosvenor Gardens, London, S.W.1.

Included in the papers selected for presentation and discussion were the following: "Mechanical Properties of Low-Carbon, Low-Alloy Steels Containing Boron," by W. E. Bardgett and L. Reeve; "The Compression Test in Relation to Cold-Rolling," by N. H. Polakowski; "Change of Electrical Resistance during the Strain-Ageing of Iron," by A. H. Cottrell and A. T. Churchman; "Plastic Strain and Hysteresis in Drawn Steel Wire," by R. S. Brown; and "The Effect of Sodium Oxide Additions to Steelmaking Slags. Part I—Use of Soda to Dephosphorise Pig Iron at 1,400° C.," by W. R. Maddocks and E. T. Turkdogan.

In addition to these single papers, there were three sets of papers on related subjects. The first series concerned the flow and mixing of gases in open-hearth and side-blown converter models and included: "Open Hearth Furnace Models. Part I—Flow Patterns in Ducts," by J. H. Chesters and A. R. Philip; "Part II—Flow Visualisation and Photography," by R. S. Howes and A. R. Philip; "Part III—Flow Patterns in Model Furnaces," by I. M. D. Halliday and A. R. Philip; "Experiments in Gaseous Mixing in Open-Hearth Models. Part I—Maerz," by R. D. Collins and J. D. Tyler; "Experiments on the Gas and Fluid Flow in a Side-Blown Converter Model," by M. P. Newby.

The second series concerned the structure of alloys of iron with nickel, aluminium, chromium and manganese, the four papers being: "Further X-ray Study of the Equilibrium Diagram of the Fe-Ni System," by E. A. Owen and Y. H. Linu; "Free Energy and Metastable States in the Fe-Ni and Fe-Mn Systems," by F. W. Jones and W. I. Pumphrey; "Microscopical Studies on the Fe-Ni-Al System. Part I— $\alpha + \beta$ Alloys and Isothermal Sections of the Phase Equilibrium Diagram," by A. J. Bradley; and "Constitution of Fe-Ni-Cr Alloys at 650°-800° C.," by W. P. Rees, B. D. Burns and A. J. Cook.

The papers in the remaining series dealt with blast furnace practice and comprised: "Investigations on an Experimental Blast Furnace," by H. L. Saunders, G. B. Butler and J. M. Tweedy; "The Distribution of Materials in the Blast Furnace. Part II—Compensated Charging," by H. L. Saunders and R. L. Wild; and "Radio-active Technique to Determine Gas Velocities in a Driving Blast Furnace," by E. W. Voice.

Simplification in Industry

THE speeding up of industry has become the dominant factor in the national effort and this is leading to more and more rationalisation and the concentration of industry, which have become the order of the day. Seeing that results are sought with the least possible delay any changes in production likely to be satisfactory economically should be undertaken as quickly as they can be applied. In this connection the recent Report* on simplification in industry is worthy of careful study. It gives the findings of a special Group appointed by the Anglo-American Council on Productivity to investigate and report upon the methods which industry in the United States has employed to limit the diversity of types and ranges of products, to consider the effect on productivity and cost and to recommend how far and by what methods the experience of industry in the United States in this matter can be applied with advantage in the United Kingdom.

There can be little doubt that greater simplification has many advantages to both producer and user. Experience during the war showed that in many sections of industry it was possible to limit the types, sizes, grades and qualities normally supplied, and changes were made which had the effect of increasing production as a whole. Much more doubtless can be done by simplification, as this report states, to ensure longer runs with fewer changes on the production line; reduced tooling and set-up time; increased mechanisation and special-purpose plant; easier training of operatives; simpler and cheaper inspection; less capital invested in idle plant, tools and space; reduction of stocks of materials, components and end products; reduced call on drawing office and design staff for special orders; simpler clerical and administrative work; easier service and maintenance; concentration of sales and advertising effort on a narrower range; and hence, increased productivity, leading to reduction in costs and prices, and to increased sales.

Simplification is a well-established practice in American industry and the Group collected evidence of the benefits derived from its application by many individual companies. There was naturally some concern as to whether the industrial user and domestic consumer had suffered as a result of this policy. The Group's conclusion is that the American consumer has been made conscious of the advantages of lower price and ready availability which flow from the deliberate policy of reduction of variety. Sales and advertising technique have had a very important influence in this connection.

It is realised that this policy is made easier in the United States because exports only amount to little more than 5% of total production and in general export markets are expected to take the standard home product. In the United Kingdom by comparison, the average exports exceed 20% of total production, while in individual industries as much as 75% of total production may be exported. And, it must be remembered, many exports are products for particular markets; the standard home products not being applicable. Nevertheless, the Group considers that a great deal can be done in our own export markets to make reductions in variety by what the Americans term "hard selling." This means that sales organisations persistently and

forcefully present to their customers, both at home and overseas, the benefits in rapid delivery and lower cost of simplified product ranges. In other words, products are sold to customers who really do not want them, but their choice is restricted and they must take their requirements from the nearest offered. In the general field of engineering, of course, the policy has the advantage of better service and spares organisation.

There is much truth in the claim that maximum production, coupled with the most economical use of materials, both raw and semi-finished, and the minimum use of man-power, can be assisted by the application of this policy; steps should be taken, therefore, to spread amongst producers and consumers a realisation of the benefit of simplification and we have no hesitation in recommending this report for thoughtful consideration.

Research and Production

HOW often are the results of research lost to the world of production? It is appreciated that in very many cases engineers who are so busily engaged with their own day-to-day problems find little or no time to study the countless research reports that are published. Therefore, it is possible that many developments in science, which would supplement the production methods at present being used, fail to find a commercial application.

To increase production knowledge the British Welding Research Association has established a team of development and liaison engineers, who are prepared, ready and able to interpret the research results of the Association, and who more than welcome the opportunity of visiting and discussing, with all those interested, any particular problem connected with the many varied applications of welding.

Mr. A. J. Hipperson, B.Sc. (who leads the team) was for many years connected with the Welding Advisory Service and has been with the British Welding Research Association since its inception. His staff includes Mr. Burt, O.B.E., B.Sc. (Eng.), who was formerly Deputy Director General (Inspection) of the Directorate General of Industry and Supplies, India, Mr. C. C. Bates who has served with the Technical Inspection Staff of the A.I.D. Ministry of Production, and Underwater Welders & Repairers, Ltd., and Mr. P. M. Teanby, one time of Joseph Lucas, Ltd., who was a member of the research staff of the British Welding Research Association prior to joining the development and liaison department.

Backing this able team are the full scientific and technical resources of the Association, including the joint B.W.R.A. and Institute of Welding Library, which presents a wealth of technical knowledge and information. It is therefore hoped that industry as a whole will welcome this new departure, and readily grasp the opportunities that are presented to it.

Let us face up to the fact that only by the operation of such liaison can industry successfully appreciate and employ the results of research with the least possible delay, so enabling every increase in productivity to be obtained. Research and industrial production must progress hand in hand, and liaison between the two must be the ever strengthening bond, thereby ensuring not only that all are availing themselves of the factual knowledge that research associations possess, but that such associations are continually kept in touch with the problems which confront industry.

*Published by the Anglo-American Council of Productivity, U.K. Section, 21, Pall Mall, London, S.W.1, price 1s.

Cold-Worked Metals and Fatigue

By Richard Saxton

Flow in cold drawing is due to the combined effect of the direct pull and circumferential pressure induced during passage through the die. Wear at the entrance of the die causes a ring formation and methods are given to eliminate this defect.

THE efficient reduction of metals by cold drawing necessitates a knowledge of crystal flow and the reaction of a particular metal to the pressure imposed, since a wide variation exists in the response to deformation by this method. Uniform flow is essential, otherwise cross-sectional stress is set up resulting in a brittle condition detrimental to further processing.

Flow should be uniform throughout section, in this respect it differs from flow in extrusion or direct strain as in tensile testing. In tensile the greatest flow takes place at the core, as may be noted from the structure on a longitudinal section formed just previous to fracture. Tensile testing is cold working by direct strain; cold drawing is cold working by lateral compression.

Flow in cold drawing is due to the combined effect of the direct pull and circumferential pressure induced during travel through the die reduction zone, and the tendency to rapid flow of the crystals at the core from the centralised pressure is counterbalanced by greater effect of the die on the outer crystals, with resultant uniform flow or grain elongation.

A striking difference in structure between a metal subjected to lateral compression and a corresponding sample stressed under direct tension is: in the former the crystalline grains are elongated into fibres, and in latter they are ruptured at the weakest point before being greatly elongated. Photomicrographs reveal, under direct strain as in tensile testing, lines of flow that are not uniform, the greatest flow being at the core. In cold working by drawing or circumferential pressure flow is more uniform.

In cold working practically all metals certain fundamental changes taken place, independent of reduction performed. First, the original equiaxed structure is transmuted to a fibrous material, and concurrently metal is hardened, and responds less plastically to further working.

Two kinds of slip take place during deformation. First, there is slip on the crystal cleavage planes, and second, slip between crystals. Research on this reaction reveals, after initial yield has taken place and metal is hardened against further yield on the same slip planes, that there is a proportional decrease in internal slip and an increase in external slip. It has been shown that the effect of cold working is to cause the various crystals to assume a practically constant orientation, and form a dodecahedral plane perpendicular to the length of material, with a cube face in surface at all points.

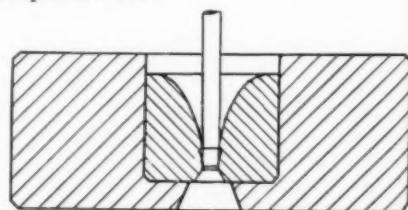
With steels, as one of the chief metals subjected to cold work in various forms, the higher the carbon and manganese content the greater the resistance to deformation. Mildness or extreme ductility under pressure, however, is not a ruling factor in reduction, as many mild-quality metals are too soft to reduce to as great a depth per draft as high-carbon steels. This is chiefly due to the lesser resistance, of milder qualities, to the stress imposed. If drafts are too heavy the resultant

effect is that of stretching of the reduced section to a diameter smaller than is furnished by the die, and the effect of stretching is a condition of differential internal stress, detrimental to further processing.

Ring Formation in Dies

The chief defect to which carbide-reducing dies are subject is wear, which occurs most frequently at the die entrance. Wear at this point is known as "ring formation" and is a sort of recess worn in the die wall in ring shape similar to that shown in Fig. 1. This, unless eliminated, is liable to form sharp edges, which break off and cut out the die. Rings invariably form at point where the material contacts the approach angle, and to eliminate without detriment to the die diameter, it is recommended to impart a 12° approach angle for a height of approximately $\frac{1}{16}$ in., which furnishes a 20°-22° angle for at least $\frac{1}{8}$ in. To work with this the entering angle should be opened to 35°.

Fig. 1.—
Cross-section
illustrating
wear ring.



If the wear is only slight it may be removed by polishing the approach angle for a short period, but if heavy it should be processed until the ring is eliminated. This will cut down the length of bearing, but the die may be worked in the same diameter until the bearing is practically worn away. Polishing of the approach angle at frequent intervals—i.e., each time the die is returned to the die room, minimises wear on the bearing, and considerable polishing may be performed without detriment to the die size.

Metal fatigue is often claimed to be a condition due to over-drawing, or when material has been worked to the elastic limit. Fatigue is not due to these factors, but is the resultant from some operation in which the material has been repeatedly overstressed. Although fatigue effects are somewhat similar to those noted in cold working, the metal is work hardened locally, becoming brittle and eventually fractures. Most metals, free from corrosion factors, will indefinitely withstand stress not exceeding a certain well defined limit, but if stress of a varying nature be applied—i.e., if the metal is subjected to frequent stress fluctuations, a stress far below the maximum static will lead ultimately to fracture.

Cold-drawn metals are, in general, an aggregate of strained grains, inhomogeneous from the view point of microstructure, and material properties. Application of stress cycles above the fatigue limit is accompanied

by a cumulative storage of energy within the metal, and a part of this is recoverable, being latent strain energy. The remainder is not recoverable, and it is generally accepted that this works to form the fractured surface or fissure at a weak part in the structure. Growth or development of this fault finally leads to fracture. The stress generated may be compressive, bend, torsional,

or a combination of these, but unlike failure by tensile stress, there is no noticeable distortion of the metal at the point of fracture, and the fractured surface invariably reveals a brittle appearance, even with metals regarded as ductile. Excessive plastic reduction or cold-working implies severe deformation, but fatigue can, and does, occur without deformation.

Recent Developments in Materials, Tools, and Equipment

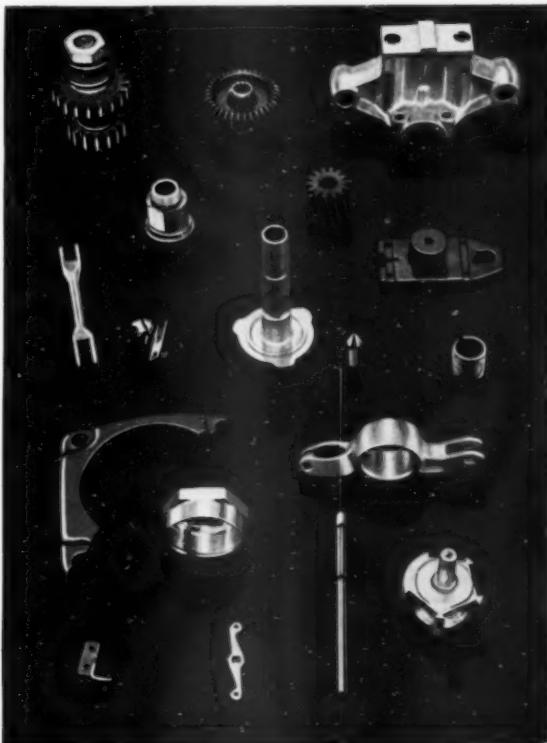
Roto-finishing

ROTO-FINISHING is a barrel-finishing method whereby de-burring, polishing, brightening and other similar finishing operations may be effected. The process is carried out in octagonal-shaped, wood-lined barrel units of the single- or multiple-compartment type, the parts being wet-treated in a mixture of mineral chips and chemical compounds. Unlike most barrel processes, there is no impact between parts as they are not carried to the top of the barrel as in most tumbling operations, but remain integral with the mass, which rises but a few degrees and then slips back due to the low speed of rotation of the machine. Relative movement does occur, however, and this is sufficient to produce the desired scouring effect. The cycle may take anything from 10 minutes to 24 hours, depending on the condition of the parts and the finish required.

The chips may be obtained in various sizes; large chips cut faster, but give a rougher finish. Chips should be of such a size that they do not wedge in any openings



Emptying Roto-Finishing machine.



Typical small parts after treatment.

in the parts. To reduce processing time and produce a superior finish the use of large and small chips in combination may be adopted.

The compound consists of alkaline and abrasive material and serves to: (1) Lubricate both chips and parts in process; (2) prevent tarnishing, etching or rusting of the work; (3) help to keep the compartment and chip mass clean; (4) provide the amount of abrasive action required to produce the surface finish desired; (5) prevent glazing or loading of chips with fine metallic particles removed during the process; and (6) correct water deficiencies. A number of compounds are available, the choice being governed by the type of finish required—dull matte, fine matte, or semi-bright.

For polishing, a softer action is required and special chips are used which are less abrasive than those used in grinding.

Britehoning is a combined polishing-honing operation which follows the polishing stage. The polished parts and chips are thoroughly rinsed in the machine and then, in order that the chips will act as a honing agent rather than a polishing medium, a special wet-honing composition is added to the mass. Parts may be britehoned directly after forming or trimming operations, provided there are no deep die and stretcher marks present. Die castings may also be processed directly when they are closely trimmed.

By the use of Roto-finishing, considerable saving of time can frequently be achieved. For instance, one CW60-2 machine will release three to four skilled men engaged on hand de-burring, grinding, polishing, buffing and other finishing work, and one man can operate eight CW60-2 machines.

Roto-Finish, Ltd., 32, Bishopsgate, London, E.C.3.

Magnetic Amplifiers for Thermo-Couples

THE magnetic amplifier, a development of the D.C. controlled A.C. choke, is not a new idea; it was known and used over twenty-five years ago. Interest was largely diverted from it by the advent of the thermionic valve, but it continued to be used in some applications such as theatre lighting control. In recent years, however, there has been a considerable increase of interest in the subject and much new research has been done at home and abroad, in which Elliott Brothers (London), Ltd. have contributed. This revival is due to improvements in magnetic materials and dry metal rectifiers and to the desire to minimise the use of sensitive relays.

For many industrial applications the rugged construction, long life and extreme reliability of magnetic amplifiers commend their use. Elliott Brothers, Ltd. have concentrated on the commercial development of these amplifiers and have standardised components, much in the same way as valves have now been standardised. They now have in production a range of three basic sizes of amplifiers, which consist of a core and windings in a hermetically sealed can. All the ancillary components such as rectifiers, bias supplies, mains transformers, etc., are provided externally, allowing maximum flexibility of circuit arrangements. As a result of this standardisation, this firm can supply complete magnetic amplifier units, quickly and economically, to suit a wide variety of applications.

One of these units which has already created an extraordinary interest, is the magnetic amplifier unit for thermo-couples. Hitherto, the means for amplifying the small signals derived from thermo-couples have invariably been complicated and expensive due partly to the difficulty of obtaining long-term accuracy. This is a point on which much of Elliott's development work has been centred with the result that the small, compact Elliott Thermocouple Magnetic Amplifier now offered maintains an accuracy of ± 40 micro-volts in spite of large variations in supply voltage and frequency. The amplifier is connected to a copper-constantan thermo-couple and operates a very robust 12-in. cirscale Elliott indicator giving full scale deflection for 100°C . A direct writing recorder can be operated *additionally* if required. Response time of the amplifier is but two seconds for full-scale deflection. Other temperature ranges can, of course, be covered if desired, and it is certain that the amplifier will find a ready reception in many branches of process control.

Chubb Safes Rust Protected A Simple and Economical Method

THERE are now few industries that do not take steps to reduce the effect of rust to which ferrous component are subject, as by so doing, the life of each product is lengthened. Frequently one of many forms of pretreatment is used before finally coating with paint or cellulose. Some of these, in addition to being inhibitors of rust, provide a good surface on the treated part, which acts as a bond for a subsequent coating of paint or cellulose.

This form of pre-treatment is often one of the processes located in the production line designed on a mass-production basis, particularly in the production of motor-car components. In the accompanying illustration, however, it will be noted that this form of surface pre-treatment is applied to fire-resisting cabinets by Messrs. Chubb and Sons, Lock and Safe Co., Ltd. This firm treat their lockers, deed boxes and fire-resisting safes in the same way, using "Jenolite" as a rust remover and neutraliser.

Method Adopted

This process is very simple, economical and time-saving. After a thorough degreasing, each part of a fire-resisting filing cabinet, for instance, is dipped into the "Jenolizing" solution, which is heated to a temperature of 55°C . After immersion, articles are placed on the adjoining draining board for a few minutes before final processing in a hot-rinse tank containing a solution of one part of Jenolite and forty parts of water, and maintained at a temperature of 95°C . This rinse speeds up production, since the temperature solution facilitates quick drying. The work is ready for the painting immediately the surface is dry, and perfect adhesion, as well as rust-proofing, results. In the case of bulkier articles, which, for obvious reasons, cannot be "dipped," the solution is brushed on, and afterwards the surface is dried with a cloth.



The pre-treatment of fire-resisting filing cabinet parts as a protection against rusting, at the works of Messrs. Chubb and Sons, Lock and Safe Co., Ltd.

Jenolite Ltd., 43, Piazza Chambers, Covent Garden, London, W.C. 2.

Reviews of Current Literature

METALLIC CREEP AND CREEP RESISTANT ALLOYS

By A. H. Sully, M.Sc., Ph.D. xii + 278 pages, with 138 diagrams and illustrations, size 5½ in. x 8½ in., and bound in black cloth, price 25s., by post 1s. extra. Published by Butterworths' Scientific Publications Ltd., Bell Yard, Temple Bar, London, W.C.2.

SINCE Tapsell wrote his book "Creep of Metals" in 1930, and summarised the knowledge then available in this field, the work performed and creep data recorded may, without exaggeration, be said to have increased tenfold. Accordingly, a further critical summary of the present position was urgently needed, and this need Dr. Sully appears largely to have fulfilled.

The book should be valuable to metallurgists and physicists as a guide to a now-recognised field of interest, and to engineers particularly as affording an up-to-date background of theory and experiment to their use of creep data in design. It is divided into seven main portions dealing respectively with, measurement of creep; creep curve characteristics; physical theory of creep and deformation; metallurgical factors influencing creep; creep of ferrous and non-ferrous alloys, and the development of creep-resistant materials.

Under "measurement of creep" a comprehensive survey is made of machines and extensometers for carrying out tensile creep tests. Some mention is made of other varieties of test, but possibly more attention might have been given to torsion, combined stress and relaxation tests, all of which have been appreciably used in recent years, particularly in more fundamental work.

The second section discusses features of the transient, quasi viscous and tertiary regions of the normal creep curve, particularly in their relation to the numerical prediction of creep properties, and records a number of the semi-empirical equations put forward with this end in view. The relation of the short and long period creep test is discussed, and some mention is made of the phenomenon of microcreep; first noted by Chalmers.

The discussion of physical theory of creep examines the various physical theories advanced in explanation of the three varieties of creep. This section is valuable in virtue of the clear way in which the merits and, more particularly, the limitations of these various theories are set forth. The engineer especially should obtain from this section, a more balanced view of the theory of the subject than can be achieved from the reading of individual papers, which are frequently misleading to readers only very partially possessed of the requisite background of physical theory.

The effect on creep of such metallurgical factors as crystal size, recovery and recrystallisation, initial plastic strain, soluble and insoluble impurities, alloy additions, heat treatment, phase changes, precipitation hardening, and service environment are considered. The information is well documented, and presents, as a cohesive whole, data which too frequently is put forward in a disjointed and factual manner.

The next two sections deal respectively with creep characteristics of carbon, low alloy, and austenitic steels, cast iron and the various engineering non-ferrous alloys. There the problem has been to present, in a reasonably digestible manner, the essentials of the very great mass of data now available, and the author has, I think, succeeded in making a well-balanced choice of

data which will serve as a sound introduction to those wishing to acquire further acquaintance with the creep characteristics of any of the particular alloys mentioned.

Finally the author makes some anticipation of the likely trends in development of future alloys required for the ever increasingly higher temperatures of operation. Suggestions are made as to the most direct and simple approach to this end. An Appendix is provided, dealing with British and American Standard Specifications for long-period creep tests.

A. E. JOHNSON.

STEEL FOUNDRY PRODUCTIVITY TEAM REPORT

By the British Steel Founders' Productivity Team. Published by the Anglo-American Council on Productivity (U.K. Section), 21, Tothill Street, London, S.W.1, or the British Steel Founders' Association, 301, Glossop Road, Sheffield, 10. Crown 4to, 108 pp. illustrated, price 3/- (postage 4½d. extra).

During March and April of this year a Productivity Team, representing the steel foundry industry, spent six weeks in the United States of America. It was the first to be sent to America under arrangements made by the Anglo-American Council on Productivity, and this Report gives an account of the Team's composition, route, observations and conclusions, and matured recommendations. Its aim is to stimulate thought and action, and to give emphasis to the ideas which made the greatest impression upon the Team during their visits to various foundries in America.

The Team was drawn from the supervisory, technical, and workshop levels in the industry and could be regarded as fully representative. In the Report it defines productivity as the ratio of the production of wealth—goods, services, and human satisfaction—to human effort expended. In applying this definition to the specific case of steel castings, although accurate measurement is not possible, the ratio of man-hours per ton (M.H.T.) may be taken as a broad indication (inversely) of productivity and on that basis the productivity in United States steel foundries is between 50% and 90% greater than in British steel foundries. Figures of productivity in American steel foundries were given by Mr. C. W. Briggs, technical director of the Steel Founders Society of America, which, in comparison with figures from the Companies represented in the Team, showed this disparity. As it could not be accounted for by marginal errors of measurement, the conclusion is inescapable that the productivity of the American steel foundry industry is markedly higher than that of the British.

The Report draws attention to the influence in America of the compulsions of fear and competitiveness against a background of constant endeavours to attain higher standards of living, and sets out the contributory means to productivity as: good factory layout and tidiness of working; use of power and widespread acceptance of machines to replace manpower; economy in the use of labour; standardisation of output; and application of the results of research.

Judged by British standards, labour in American steel foundries is conspicuously unskilled and trades union organisation is not on a craft basis. Great economy is exercised on the use of skill. The careful servicing of skilled men, so as to concentrate their energies on skilled operations, was one of the most important observations made. It should not be assumed, however, that the American foundries are practically

devoid of skilled craftsmen, but that the exercise of skill has been taken from the shop floor into the pattern shop, the methods office and the brains of the supervisors. American foundries have organised the making of steel castings in such a way that skilled moulders are not required except for a vanishing percentage of the work. Even short runs—as few are eight off on pattern—are mounted for machine moulding. Skill, in the sense of ability to plan the production methods required to produce sound castings without wastage of materials, remains very much in evidence.

In metallurgical practice the trend is towards producing steels to meet physical test specifications, rather than analytical limits. The quality of American steel castings is satisfactory and functionalism is the criterion of satisfaction. The adoption of non-destructive testing is widespread. It is noteworthy that acid electric melting is in general use in America and power inputs are applied that are twice as great as in equivalent arc-furnace installations in Britain. The good quality scrap available enables the employment of this economical technique.

Managements are actuated by the belief that high rates of production are essential to individual and collective success. Their aim is the maximum output from the minimum labour force. This aim is developed by labour saving, reduction of the grade of labour required, together with maximum mechanisation and control of sales to encourage a higher order of repetitiveness, combine with a careful attention to maintenance of mechanical plant and a willingness to try new methods. The belief that high rates of production are essential is held by the whole body of American industrial thought and at all levels.

The Report submits two questions: Is high productivity really desired by British steel founders? Over what features in the British industrial system does the desire for high productivity take preference? It insists that high productivity must be established as a touchstone of industrial conduct. This Report should be carefully studied by all engaged in this important industry.

W.A.

ELASTICITY AND ANELASTICITY OF METALS

By C. M. Zener, 1948, 170 pages, 56 illustrations and diagrams; Cambridge University Press, Bentley House, 200, Euston Road, London, N.W.1, and in the United States of America, University of Chicago Press. Price 22.6 net.

THIS book, the first monograph published by the Institute for the Study of Metals in the University of Chicago, describes the work of Zener, together with development generally on internal friction, or, elastic after-effect, damping capacity, etc. (or, the other terms which now cover the phenomenon manifested by deviations from Hooke's Law). Dr. Zener is a distinguished mathematical physicist and his treatment is based throughout on an intensive mathematical background. Thus, a great part of the book is beyond the understanding of "the ordinary metallurgist," but the descriptive portions are in the main of considerable assistance to those interested in physical metallurgy—a state of affairs only too rare in authoritative dissertations of this kind.

The author deals first with the elasticity of metals—stress and strain concepts; effect of crystal symmetry; elastic constants and their dependence on temperature; and "microelasticity"—the relation between energy and

strain of any elementary region which passes from one configuration of minimum energy to another by a homogeneous strain so that plastic deformation will occur below the theoretical limit.

The greater part of the book is devoted to the theory, measurement and physical interpretation of anelasticity, described by the author as the term chosen to denote "that property of solids in virtue of which stress and strain are not single-valued functions of one another in that low stress range in which no permanent set occurs and in which the relation of stress to strain is still linear." The various types of anelasticity are then discussed. The origin of these anelastic effects is now understood to a greater extent than formerly and this has led to their better control.

The practical physical metallurgist—in close touch from day to day with the applications of physical metallurgy, but not always able to keep abreast of current physical developments of a fundamental nature—will find this book "heavy going." He will be attracted, on the other hand, by certain features, notably the discussions on the nature of grain-boundary material and on the confusion which has arisen between the theories of Rosenhain and Beilby. Dr. Zener (p. 148) stimulates thought on this controversy when he points out: "No controversy need arise, once it is realised that it is not necessary for any portion of the metal to be amorphous in order that the grain boundaries may behave in a viscous manner . . ."

Physical metallurgists will await with interest the publication of further Monographs of this standard. All will envy the quality of presentation and the excellence of printing and publication which American readers are able to enjoy.

K. G. LEWIS.

Questions and Answers on Zinc Alloy Die Castings

AN interesting and informative booklet has recently been issued by the Zinc Alloy Die Casters Association. It is addressed to potential users of zinc alloy die castings and endeavours to anticipate some of the questions which may occur to them. For example, it shows how the process can be used economically for short runs as well as for long runs and for making small as well as large parts. The illustrations have been carefully chosen from a great number of possible examples, and each shows a particular aspect of zinc alloy die casting which might otherwise escape the notice of all but the experienced user. Copies of the booklet can be had on request to the Association whose address is: Lincoln House, Turl Street, Oxford.

Licences for Cold Pressure Welding

WORLD-WIDE interest has been created in the Cold Pressure Welding process developed by the General Electric Co. Ltd. in its research laboratories. The Company has now decided that since this method of welding can be put to a wide range of uses, both inside and outside the electrical field, industry as a whole should have access to the process, so that the technique can be exploited and developed in the widest possible way. Arrangements have accordingly been made for the issue of non-exclusive user licences to all firms who wish to use the process. Further information on application to Magnet House, Kingsway, London, and quoting the reference PUBLIJHJ.

METALLURGICAL DIGEST

High Strength Nickel Alloy Retains Properties at High Temperatures

By C. A. Crawford

UNUSUALLY high strength at ordinary temperatures and at red heat is obtained by suitable heat treatment of Inconel X, a recently developed high-temperature alloy. It is claimed to be that latest achievement in the search for alloys suitable for gas turbine parts, which require high strength and low plastic flow at temperatures up to or above 927° C. Resistant to chemical corrosion and oxidation this alloy is the most recent of the series of age-hardenable alloys evolved from Inconel.

The decrease in tensile and yield strengths of this new alloy, from room temperature up to 593° C. is very small, 90% of the tensile and yield strengths of the directly aged material being retained up to 482° C. and 80% up to 593° C. When the material is solution treated and aged to obtain optimum properties above 593° C., the yield strength at 593° C. is 90% of the room temperature value.

The high strength of the alloy is the result of composition and heat-treatment and does not depend on hot working below the recrystallisation temperature. Heat treatment affords more uniform hardening from surface to centre of large sections than can be obtained by the "cold forging" method referred to. Cold finishing can be used to advantage for moderate temperature applications below 593° C., for these applications highest strength is obtained by ageing directly after hot or cold work with an inter-

mediate stress equalising anneal for large sections. For applications above 593° C., and where loads are to be sustained for long times, the useful strength of Inconel X is obtained only by full heat treatment. Above 816° C., the effect of age hardening is lost and the alloy is used in the annealed condition.

Comparative oxidation tests indicate that the oxidation resistance of Inconel X is of the same order as for the standard alloy at temperatures up to 871° C. Above its resistance is less than that of standard Inconel. Production experience indicates that the new alloy can be forged, welded, and machined successfully; no unusual difficulties have been encountered. The composition limits of the alloy are given as:

Nickel	70.00	min.
Chromium	14.00-16.00	
Titanium	2.25-2.75	
Columbium	0.70-1.20	
Aluminium	0.40-1.00	
Iron	5.00-9.00	
Manganese	0.30-1.00	
Silicon	max. 0.50	
Copper	max. 0.20	
Carbon	max. 0.08	
Sulphur	max. 0.01	

Applications

The exceptional strength of Inconel X at all temperatures from subzero to 593° C. make it especially useful where corrosive or oxidising conditions exist under stress. It compares in strength with heat treated steels and retains its strength to temperatures beyond the

drawing temperature of steels suitable for high temperature steam applications. Because of its high strength from subzero temperatures up to and beyond present day steam temperatures, the alloy is particularly adapted to bolts, springs, and all types of forgings or fabrications for supporting either static or dynamic loads where chemical attack renders simpler alloys unusable.

It is used for gas turbine rotor wheels, gas turbine blades and vanes. Bolts of this new alloy have been used successfully to fasten the assembly of outside supporting structures and housings of the combustion system in aircraft gas turbine engines, and in assemblies of multiple-stage turbines or other holding devices that operate at elevated temperatures.

Several laboratories have used it for specimen holder grips in tensile, stress rupture, creep and similar tests conducted at high temperatures. Because the methods employed require that the specimen holders remain in hot zones for the period of testing, which is frequently long, the temperature of the grips becomes almost as high as that of the test specimens.

This new alloy is expected to be useful at all temperatures up to 343° C. as a particularly strong non-ferrous spring material. The properties of the alloy as a spring, from 343-510° C. indicate future usefulness in this band of temperatures where little else is available except the tool steel type of alloy. Springs of this material are also expected to have some use at higher temperatures up to 649° C. and possibly to 760° C.

In sheet form, the new alloy has been used in parts of jet engine and rocket-motor assemblies where stresses higher than can be imposed on standard

From *Materials and Methods*, Oct., 1949, 57-61.

PHYSICAL AND MECHANICAL PROPERTIES

Temperature, °F.	Instantaneous Coefficient of Expansion (in. per in. per °F. × 10 ⁶)	Modulus of Elasticity × 10 ⁶		Impact Strength (ft.-lb.)		Fatigue Strength (Psi)	
		Tension	Torsion	Solution Treated, 2,100° F., 2 hr. Aged, 1,550° F., 24 hr. Aged, 1,300° F., 20 hr.	Hot Rolled Aged 1,300° F., 20 hr.	10 ⁴ Cycles	10 ⁸ Cycles
-220	—	—	—	35	34	—	—
-109	—	—	—	26	37	—	—
80	—	—	—	—	—	—	—
400	8.0	—	—	42	44	—	—
870	8.4	—	—	50	46	—	—
1,200	10.0	23.0	8.1	45	45	67,500	55,000
1,500	11.4	21.0	—	49	49	54,000	49,500
1,700	12.4	18.5	—	67	52	47,500	36,000
1,750	12.8	—	—	113	82	—	—

Inconel are involved. Members designed to function as springs, including expansion bellows, have been cut or formed from sheet. Strip, down to light gauges—0.006, 0.008, 0.010 in., etc.—is being used as spring material in valves of resonant jet engines.

Physical and mechanical properties of this new alloy are reproduced in the accompanying table, but the heat treatment of the material in various forms is also tabulated, as are the hardness ranges resulting from mill operations and heat treatment. Curves

are given showing the strength of this alloy at various temperatures and different heat treatments and an informative creep curve. Considerable information is also given on the fabricating properties of the alloy.

Designing with Beryllium Copper Casting Alloys

By John T. Richards

THE place in industry of wrought beryllium copper (strip, rod and wire) has been well established. Less, however, has been reported concerning cast beryllium copper, although age-hardened castings deliver the same useful properties as obtained in wrought forms. This means that cast parts, depending upon the alloy selected, offer tensile strengths up to 85 tons/sq. in. or hardness to 450 Brinell, or electrical conductivities to 50% that of pure copper.

In addition to high strength and good conductivity, the following characteristics of beryllium copper may be of interest to materials engineers:

1. Non-magnetic.
2. Good resistance to wear and corrosion.
3. Superior elevated-temperature properties as compared to other copper-base alloys.
4. Retention of ductility in sub-zero atmospheres preventing loss of impact strength or low-temperature embrittlement.
5. Good fluidity in the molten state resulting in maximum detail in "as-cast" parts with minimum machining.
6. Relatively low pouring temperature facilitating foundry handling.

From *Materials and Methods*, Sept., 1949, 70-73.

TABLE I.—NOMINAL COMPOSITION OF SOME BERYLLIUM COPPER CASTING ALLOYS.

Beryllium	2.00-2.25	2.60-2.85	0.55-0.70
Cobalt	0.35-0.60	0.35-0.65	2.35-2.60
Copper	Remainder	Remainder	Remainder

TYPICAL PHYSICAL PROPERTIES OF THESE ALLOYS IN THE FULLY HEAT TREATED CONDITION.

Specific Gravity	8.09	8.09	8.61
Density (lb. per cu. in.)	0.292	0.292	0.311
Magnetic Properties	non-magnetic	non-magnetic	non-magnetic
Melting Range	1585-1750	1585-1750	1825-1950
Modulus in Tension, Psi.	19,000,000	19,000,000	18,000,000
Average Coefficient of Linear Exp. (per °F.) " " " (per °C.)	0.0000095 0.0000170	0.0000093 0.0000166	0.0000098 0.0000177
Thermal Conductivity (Btu. sq. ft. ft. /hr. /°F.) (Cal./sq. cm. cm. sec. /°C.)	67-75	59-69	133-150
Shrinkage (in. per ft.)	0.27-0.31	0.24-0.28	0.54-0.61
	3/16	3/16	3/16

techniques follow those developed for other alloys.

Beryllium copper casting alloys have excellent casting characteristics and are finding increased applications in many fields. In comparison with steel they offer superior corrosion resistance and conductivity, and compare favourably in strength, hardness and wear resistance. Moreover, beryllium copper gives better detail, smoother surfaces and closer dimensional control. Compared to other copper-base alloys, beryllium copper parts are stronger and give better resistance to wear and moderately elevated temperatures.

Useful processing information is given including machining, heat treating and cleaning.

Mechanical Properties of Forging in Steels Containing Low Percentages of Nickel, Chromium and Molybdenum

By R. POTASZKIN

THE mechanical properties of three structural steels containing small quantities of nickel, chromium and molybdenum, the compositions of which corresponded to those of American National Emergency Steels Nos. 8627, 8735 and 9945 were determined on forged pieces, 25, 50, 100 200 mm. in dia. after various heat-treatments. The results are presented graphically. The effect of mass was to decrease the elastic limit, tensile strength and ratio of the former to the latter with increasing section, particularly in bars of diameter between 25 and 50 mm. The variations of the elongation and reduction of area were not pronounced above a certain section. The mass effect was more accentuated after oil-quenching than after water-quenching and decreased with increasing tempering temperature. The maximum section for each steel can be quenched without cracking in water in the ordinary way or by a retarded

From Societe Francaise de Metallurgie, Oct. 20, 1948, *Revue de Metallurgie*, Memoires, 1948, 46, Mar., 125-140. Abstract from *Jour. I. S. Inst.*, Aug., 1949.

treatment and the thermal treatments producing the highest elastic limit and toughness values are given. The best results were obtained by quenching small bars in oil and larger ones in water. The "retarded" water-quench was very suitable for the steels with

higher carbon contents. By using certain conventions, the three steels are compared with Cr-Mo, Ni-Cr-Mo, Cr-Mn and Cr-Mn-V steels, and classified in relation to these latter according to their elastic limits and toughness for equivalent tensile strengths.

Creep Tests on some Unalloyed Basic-Bessemer Steels and High Alloy Heat Resisting Steels at 500° and 900°C.

By A. POMP AND A. KRICH

CREEP tests in the 500-900°C. range lasting over 1,000 hrs. were made on three plain carbon steels (C 0.04%, 0.21%, and 0.23%), three chromium steels (Cr 6%, 18%, and 24%), and 19/14, 22/6, and 25/18 austenitic chromium-nickel steels. Of the many methods used to evaluate the creep strength, the following three were considered most suitable:—(a) The load which produced a strain rate of $1 \times 10^{-4}\%$ /hr. between 900 and 1,000 hr.; (b) the load causing 0.2% permanent elongation in 1,000 hr.; (c) as a short time test, the load producing a strain rate of $3 \times 10^{-4}\%$ /hr. between 35 and 100 hr. These three methods gave creep strengths for the unalloyed steels of 4-8 kg./sq. mm. at 500°C. and only 0.35-3 kg./sq. mm. at 600°C., but no explanation of the difference was found. The 6% and 18% chromium steels decreased in creep strength from 2 kg./sq. mm. at 600° to 0.5 and 0.7 kg./sq. mm. respectively at 800°C., whilst the 24% chromium steel had a somewhat lower creep strength at 700°C. than the steels with less chromium. Of the austenitic steels the 19/14 steel at from 700°-900°C. (8 and 1.8 kg./sq. mm. respectively) was much superior to the others. The 25/18 steel at 800°C. was no better than the chromium steels. There was evidence that, at low loads, tests of very long duration are necessary. Time-elongation curves plotted on a double logarithmic scale are parabolic for only a short period, and quite false values may result from extrapolating the data from 100-hr. tests to 1,000-hr.

From *Mitteilungen aus dem Max-Planck Institut für Eisenforschung, Report No. 490, Archiv für das Eisenhüttenwesen*, 1949, 20, Mar.-Apr., 125-134. Abstract from *Jour. I. S. Inst.*, Aug. 1949.

STEELS IN GERMAN AIRCRAFT

VOLUME II is now ready and some copies of **Volume I** are still available. Order direct from The Kennedy Press Ltd., 31, King Street West, Manchester, 3, Price 10s. 6d. post free for each volume.

From *Jour. Res. Nat. Bureau of Standards*, RP2013, 48, No. 2, 1949.

Influence of Strain Rate and Temperature on the Creep of Cold-Drawn Ingot Iron

By William D. Jenkins and Thomas G. Digges

A STUDY was made of the effects of variations in both strain rate and temperature on the creep characteristics in tension of cold-drawn ingot iron. The iron was initially in the form of a $\frac{1}{4}$ -in. round bar as cold drawn approximately 13% reduction in area. The chemical composition of the ingot iron used was as follows:—

C	Mn	P	S	Si
0.019	0.029	0.005	0.018	0.005
Cr	Cu	O ₂	N ₂	H
Nil	0.05	0.08	0.0004	Nil

Tension tests were made at different strain rates and temperatures on this ingot iron. Some of the creep tests at 600°, 700° and 800°F. were discontinued after the beginning of the third stage, and these specimens were subsequently tested at room temperature. In another series of tests in which the specimens were carried to complete fracture at 500°, 600°, 700° and 800°F., the strain rate during the third stage was maintained nearly constant and equal to that in the second stage by repeatedly adjusting the load during the third stage. Hardness measurements at room temperature and metallographic examination were made on specimens representative of the initial material and after testing in creep under the varying conditions used.

The relationship between true stress and the creep rate in the second stage was not linear when either the stress or the logarithm of the stress was plotted against the logarithm of the strain rate. The use of a linear relationship in either of these plots for extrapolating to very slow strain rates is not justified.

The resistance to creep in the second stage and the extension at complete fracture at 700°F. were materially affected by variations in rate of loading in the first stage.

Although variations within the range of strain rates used in the creep tests

at 700° and 800°F. had no significant effect on plastic extension at the beginning of the third stage, evidence obtained in tests at 500° and 600°F. supports the belief that this extension is markedly increased with sufficiently high strain rates. The strain rates required to produce relatively high plastic extension at the beginning of the third stage were affected by the test temperature in that the high ductility was obtained at slower rates as the test temperature was decreased.

The third stage of creep was initiated without necking of the specimens, and the results of tension tests made at room temperature on specimens previously extended into the third stage at different temperatures showed that no general deterioration or microcracking of the specimen occurred up to the time this stage was reached. Regardless of strain rate or temperature used, however, considerable necking occurred during the third stage of each specimen tested to complete fracture.

The general trend was for the ductility (plastic extension and reduction of area) at fracture to increase as the strain rate increased. The plastic extension at fracture decreased with an increase in test temperature, but in the creep test no consistent relation was obtained between temperature and reduction of area at fracture.

The resistance to both creep and fracture increased as the temperature was decreased. The stress at fracture also increased as the strain rate increased.

The fractures were predominantly transcrystalline in the tension tests with the different strain rates used at and below 600°F. and intercrystalline at test temperatures of 700° and 800°F.

Suspended-abrasive Media used with Blast Cleaning

By Edward Rosen

BLAST cleaning with suspended-abrasive media is receiving growing attention as a modern method of mechanical finishing. The method is a controlled process which consists in the use of a circulating abrasive suspended in an aqueous solution. This spray, under pressure, is directed by means of a nozzle against the metal. By controlling such mechanical variables as abrasive mesh size, type of abrasive, ratio of abrasive to solution, air pressure, angle of infringement, and gun distance, it has become possible to produce a wide range of distinct satin and matte finishes. In addition to this variety of decorative effects, this method of blasting offers many other advantages and is being employed for numerous purposes ranging from the treatment of dies and tools to the final finishing of the end product of the dies and tools.

One of the original processes involved the use of ordinary dry sand from a standard type sand blast generator which fed the sand to a special gun and nozzle where a metered flow of water was mixed with the sand under air pressure. Continual refinements of the process led to the development of numerous corrosion inhibitors which, when added to the solution, retarded oxidation on the metal surfaces being blasted. Intensive experimental work has been carried on with various abrasives. A siliceous rock, known as Novicite, which is available in mesh sizes of 100 to 5,000 has been found to work well for this type of blasting. In addition, the availability of versatile cabinet equipment in which to carry out the operation has made the process applicable to a wide variety of jobs. The equipment used for liquid blast cleaning includes a water-tight cabinet provided with a hopper tank for mixing, storing and re-collecting the suspension, a circulating pump, and a blasting gun equipped with two lengths of flexible hose, one for abrasive feed and one for air supply. The cabinet has vision windows, loading doors, air control valves, and a ventilating system to remove the fog created by the blasting. The operator works from the outside of the cabinet and places his arms through gauntlet-covered armholes. The work to be finished is held in one

hand or rested on a work table while the other hand directs the blasting stream.

Of the numerous advantages cited for this method, the following appear most important at this time:—

1. Produces a very smooth surface with a non-directional finish that appears finely etched.
2. The minute etching effect of the abrasive honing causes the surface to hold and distribute lubricants evenly.
3. The peening action has the effect of cold working the surface of the metal.
4. It will hone surfaces when tolerances are as low as 0.0001 in., and will reach crevices in intricate parts which are hard to deburr and polish by other methods, maintaining contour control.
5. Economical and rapid.

6. Simplicity of control and operation.

Because the spray effect permits uniform removal of material from contoured surfaces and is effective in deburring internal fillets, edges, and corners that are virtually impossible

to clean, deburr or finish by other means, this process has been widely used by the aircraft industry for engine overhaul, propeller maintenance, paint removal and new parts manufacture. Supercharger impeller blades, gear teeth, splines, connecting rods, guide vanes, threaded parts and various instrument details necessitating close tolerance finishing or cleaning have been successfully treated on a production basis. Liquid blasting of aircraft engine cylinders which have been porous-chrome plated has shown reduction in scuff and wear polish because the treatment opens up secondary lubrication channels, thereby increasing surface area for lubricants. In addition to effectively removing electrolytic debris from bearing surfaces, it also eradicates any grinding or polishing lines which otherwise might serve as natural drainage routes for the lubricant. The matte finish itself provides a good lubrication surface, since it is nothing more than a mass of microscopic craters which will retain oil beads capable of sustaining a great compression load. In cylinders thus treated, there has been reported by engine manufacturers results indicating less oil consumption, less wear polish of the cylinder walls, and faster running-in time.

Room Temperature and Artificial Ageing of an Aluminium-Silicon-Magnesium Alloy

By G. Gürther and H. Weigelt

EXPERIMENTS are described which were carried out on an alloy having the composition, 9.5% silicon, 0.57% magnesium, 0.3-0.4% manganese and 0.3-0.4% iron, the remainder being aluminium. Specimens were quenched after solution heat treatment at 530°C. and then aged at room temperature (20°C.) and at 100°C., 150°C., 200°C., 250°C. and 300°C. for periods up to 9 months. The effects of ageing were studied by means of hardness tests and microscopic and X-ray (lattice constant) examinations. A study was also made of the phenomenon of reversion, i.e., the effect produced by heating a specimen substantially fully aged at room temperature or at one of the lower ageing temperatures for a short period at a higher temperature.

It was found that at temperatures up to 100°C. the increase in hardness

was still continuing after 9 months. At 150°C. a maximum hardness was reached after about 2 days and was followed by a slow falling off. The maximum hardness is higher the lower the temperature at which ageing takes place.

Microscopic examination reveals the precipitation of Mg₂Si only after the hardness had started to decrease. No evidence could be obtained from lattice constant determinations by X-rays.

Reversion of lower temperature age hardening becomes noticeable at temperatures above 150°C., the effect being different depending on the stage reached by the lower-temperature age hardening process. In the early stages of the process reversion is accompanied by an initial increase in hardness, the drop in hardness sets in after a time which is shorter the longer the lower-temperature age hardening process has been and the higher the temperature at which reversion is effected.

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
INSTRUMENTS AND MATERIALS

This Supplement

Announced last month, with this first number of a new volume of *METALLURGIA*, we commence publication of our Laboratory Methods Supplement. The subject being largely a factual one, we do not propose regularly to burden readers with our opinions on this page, although occasions may arise from time to time when we shall wish to do so. In this first issue, for instance, we feel it incumbent upon ourselves to outline, for the benefit of readers and contributors alike, the ideas we had in mind in embarking on this venture.

The number of published papers and articles on subjects of a metallurgical interest has increased enormously in recent years—so much so, in fact, that a stage has been reached when it is becoming increasingly difficult for the metallurgist to note, let alone read, all the available literature. What reasons, then, can we advance for adding to this ever-widening flood? In the first place we believe that the value of experimental work should not be underestimated. Probably as a result of limitations of space, there is a tendency, in published papers, for the experimental technique to be dismissed rather summarily, but this should not be taken as a measure of its importance. In one way or another, the metallurgist is dependent on experimental work in making decisions on his day-to-day problems, and large numbers of scientifically trained individuals are regularly engaged in such work, whether of a routine or research nature. In the course of their labours considerable time is often spent in devising methods, and in designing and making equipment when, not infrequently, methods of attack on the same, or a similar, problem have already been successfully developed elsewhere, but have not seen the light of day as far as publication is concerned.

It is with a view to the rectification of this state of affairs that we throw open these columns for the publication of articles or short notes in which the main consideration is the method employed rather than the results attained. In the main, such publication does not involve any question of industrial secrecy and we are of the opinion that, in providing a medium for the interchange of such information, we shall, by reducing multiplicity of effort, be contributing to the effort to achieve economy in scientific man-power.

It should be appreciated that the term "laboratory" in the title of this supplement is intended to be used in its broadest sense. Obviously, such operations as, for instance, the metallurgical inspection of large forgings by specialised techniques, cannot be carried out in the confines of the laboratory itself, and it is our intention that the field covered shall include all the practical operations performed by the metallurgist and those associated with him on the chemical and physical sides. In a leading article in the first issue of this Journal, just twenty years ago this month, the view was expressed that the metallurgist is essentially a chemist. The intervening years have seen some shift of emphasis from

chemistry to physics, and even in such predominantly chemical operations as analysis there is an increasing use of physical methods at some stage in the proceedings. Nevertheless, the metallurgist remains something of a mixture of chemist, physicist and engineer, a fact which will be reflected in these pages.

Among the subjects we propose to include in this section are metallography; mechanical testing; chemical analysis, including micro-analysis; X-ray crystallography; all forms of non-destructive testing; instrumentation; new equipment; laboratory news, etc. From time to time, new and interesting metallurgical laboratories will be described. It is also hoped to include a section in which reference will be made, by means of digests, abstracts or at least, mention, to work in these fields published elsewhere. Obviously it will not be possible to deal with each subject in every issue, but it will be our endeavour to maintain a balance over a number of issues.

This is essentially a contributed section, as the information published must emanate from the laboratories themselves. The articles need not be lengthy; in fact, the shorter the better, provided brevity is consistent with completeness. There is one point in this connection which is worthy of special emphasis. In order that a description of a method shall be of value to other workers, it is absolutely essential that due importance should be attached to difficulties and snags. At some time or other, most of us have experienced the difficulty associated with following a method which is described in insufficient detail or in which the snags are glossed over. One then has to spend a considerable amount of time in overcoming teething troubles which could have been avoided by adequate attention to detail in the first place. It is our hope that authors will bear this point in mind when preparing their contributions.

In most fields of human activity there is room for differences of opinion and it may be that readers will, as a result of their own experience, have views which differ from those expressed in our published articles. Our correspondence columns are available for the clarification of such issues, and we hope that advantage will be taken of these facilities.

As those who have visited the Exhibitions arranged by the Physical Society and the Scientific Instrument Manufacturers Association will appreciate, developments are constantly taking place in equipment and instruments suitable for use in the laboratory. It is our intention to bring to the notice of readers those which are likely to find application in the metallurgical field, so that advantage may be taken of their existence in the design of new apparatus and in the equipping of new laboratories. We therefore welcome information regarding such new equipment, particularly where it contains technical details of value to the potential user.

In conclusion we would repeat that this is *your* Supplement—it is our wish that you should make use of it to the full.

Metallurgical Education in Norway

By A. B. Winterbottom

(Reader in Metallography, Technical University of Norway)

As a result of her large water-power resources, coupled with her lack of coal, the Norwegian metallurgical industry is largely of an electrometallurgical character. This fact is, naturally, reflected in the training given to metallurgical students in the Technical University of Norway, at Trondheim. That other aspects of the subject are not neglected will be seen, however, from this article which includes details of the new Metallurgical Institute now nearing completion.

ABRIEF survey of the development of metallurgical education in Norway is not inappropriate in view of the Iron and Steel Institute's meeting there in June and the building of the new Metallurgical Institute at Trondheim, now nearing completion.

The contrast in the development of the metallurgical industries in Norway and Sweden is very striking and not completely explainable in terms of different natural resources and populations. So far the emphasis in Norway has been almost exclusively on process metallurgy, the production of metallic raw materials: ferro-alloys, pig-iron, sulphur, aluminium, nickel, copper and zinc. Sweden has in comparison less extractive metallurgy but, as all know, a considerable high-quality steel production and a highly developed engineering industry. It is interesting to note that Jernkontoret (the co-operative organisation comprising iron and steel-making firms in Sweden which celebrated its two hundredth anniversary last year) has conducted both technical and fundamental metallurgical research for many years at its Metallographic Institute directed until recently by Carl Benedicks.

Furthermore, both process and physical metallurgy have had a number of distinguished exponents at the Royal Technical Institute in Stockholm. Names such as Hultgren, Borelius, Westgren, Phragmen, Wiberg, Bo-Kalling are known throughout the metallurgical world.

In Norway metallurgical education and research has been on a much more modest scale. When Norges Tekniske Høgskole (the Technical University of Norway) was established in Trondheim in 1910, Vogt was called to the professorate of geology, mineralogy and metallurgy. Haakon Styri was reader in the metallurgy of iron and steel from 1914-1917.

In 1920, however, an independent chair in metallurgy was set up and Harald Pedersen became the first professor. Pedersen was interested commercially in a variety of metallurgical enterprises and his name is associated with an alternative process to the Bayer method for producing pure aluminium oxide from



Fig. 1.—General view of the new Institute.

bauxite. While lectures were given in the University, Pedersen fitted out laboratories at one of his ferro-silicon plants in Trondheim for laboratory training and research. Pedersen's interests and teaching were almost entirely of a process metallurgical character. He died during the war and the vacant chair was first filled in 1946 by the appointment of Robert Lepsøe, who, for many years, had been directing research at Consolidated Mining and Smelting Company, Trail, B.C.

Plans for extensions and additions to the Technical University had been under consideration, during the war, by a professor of architecture. These gave the neglected Metallurgical Institute a high priority and work on the new building was started in 1947. At the same time the present author was appointed dosent (reader) in metallography but was not able to take up the post until the following year. A general view of the new Institute which is now practically completed is shown in Fig. 1, while Fig. 2 is a plan of the buildings. Essentially it consists of a large hall for pilot scale work with one parallel saw-tooth bay for transformers and converting equipment, another similar bay for laboratories, and a three-storey block along the side of the foregoing for offices, library and some smaller laboratories.

The new Institute aims at providing accommodation for: (1) The training of up to about 10 metallurgists each year; (2) laboratory investigations in process and physical metallurgy; and (3) pilot-scale plant studies for Norwegian industry of a process metallurgical character.

The staff projected and in part appointed consists of:—

1. *Teaching*: One professor of metallurgy with two assistants; one reader in metallography with one assistant.

2. *Research*: Research fellows attached to the professor and reader, as necessary for research projects, up to a maximum of about eight.

3. *General*: Secretary, workshop superintendent, mechanic and laboratory assistant.

The laboratories now being equipped are as follows:

Process Metallurgy Laboratories (117 and 118)*.—Two similar laboratories provided with concrete floors, fume cupboards, fume hoods for small furnaces, and bus bars for A.C. and D.C., up to 150 V., 4,500 A. and 2×20 V., 500 A., respectively.

Hall for Pilot-scale Work (121) (380 sq. m.), with 3-cwt. Birlee Lectromelt single-phase furnace of 150 kVA.; space for single-phase furnaces up to about 500 kVA. maximum, 10-90 V., 12,000 A., supplied from

a transformer installed in the adjacent machine bay (120); space for fused salt electrolysis or wet electro-metallurgy with D.C. power up to 10 V.-10,000 A. from a Canning generator in the adjacent machine bay, as well as for general roasting and smelting operations with oil or gas firing, if necessary, and for wet process metallurgy.

Melting Laboratory (119), with 50 kg. open and vacuum A.S.E.A. high-frequency furnaces supplied from a 70 kVA. motor generator in the machine bay for experimental melting and casting, if necessary, under reduced pressure. A 15 kw. Erco spark-gap high-frequency generator for smaller experimental melts and for hot extraction analyses is also installed here.

Materials Working and Testing Laboratory (05). Here the equipment is for teaching and research, since ordinary specification testing is catered for by the test house in the main building of the University. It includes hand rolls (grooved and plain); wire-drawing equipment; hardness tester (Firth Hardometer with diamond pyramid) miniature tensile testing machine (Hounsfield tensometer); four endurance testing machines (Wöhler type); and space for setting up creep and stress rupture equipment, electric muffle and air circulation furnaces for heat-treatment; and Jominy end-quench hardenability test apparatus. Salt-bath furnaces for heat-treatment are installed in the process metallurgy laboratory.

Microscopical Metallography Laboratories (115, etc.), include a cutting-off, mounting and grinding room with a multi-spindle machine for grinding on lead or wax laps; a polishing and etching room with automatic and non-automatic polishers (Cooke Troughton & Simms); and a microscope room with working places for five bench microscopes. Three of the bench microscopes are of the latest type of Cooke Troughton & Simms general purpose metallurgical microscope, with mechanical stages added. A Reichert universal camera microscope "MeF," a Vicker's Projection Microscope and photomacrographic equipment are also installed here. The dark room with usual equipment is adjacent to the microscope room.

X-ray Metallographic Laboratories (08 and 09). These accommodate a demountable gas tube set (Baird

First Floor :
 202—Analytical Laboratory.
 204—Library.
 206—Institute Office.
 208—Dosen A. B. Winterbottom.
 210—Special Research Laboratory.
 212—Laboratory Supervisor.
 214—Assistant.
 216—Professor Robert Lepsoe.

Ground Floor :
 103—Workshop.
 105—Stores.
 111—Steward.
 112—Lecture Theatre.
 113, 114—Physical Metallurgy.
 115, 115A, 115B and 115C—Microscopical Metallography.
 117, 118—Process Metallurgy Laboratories.
 119—Melting and Heat-treatment.
 120—Transformers and Conversion Equipment.
 121—Pilot-scale Process Metallurgy.

Cellar :
 01—Calorifier, etc.
 02, 03—Stores.
 04—Spectrographic Analysis.
 05—Mechanical Working and Testing.
 07—Corrosion.
 08, 09—X-ray Metallography.

Fig. 2. Showing the general layout of the Institution.

Associates, Cambridge, Mass.), a sealed-off tube set (Machlett) and a Metropolitan-Vicker's "Raymax" continuously evacuated demountable tube with space for the addition of a Geiger counter set later. Cameras include: Unicam high-temperature 19 cm. and ordinary 9 cm. powder cameras and a "Raymax" back reflection camera. A number of small powder cameras and a crystal monochromator will be added later.

Physical Laboratories (113 and 114)—these two small laboratories are available for pyrometric standardisation, cooling curves, electric resistance and magnetic measurements, dilatometry and for laboratory scale isothermal treatments.

Analytical Laboratories (202 and 04), include a small chemical laboratory with balance room equipped for gravimetric, volumetric, electrometric, and absorptiometric methods of analysis of metallic materials (hot extraction analysis is carried out in the melting laboratory) and a spectrographic laboratory with a large Hilger quartz spectrograph and associated comparator and spectro-photometer.

Special laboratories include a small corrosion laboratory (07) and one for polarimetric study of surface films (210).

Other facilities include a workshop (103) with lathe, shaping machine, large and small pedestal drilling machines, power saw, grinding wheels and electric and gas-welding equipment; store rooms (105, 02 and 03); wash and douche room; refreshment room; library with places for 12 readers (204); general office (206); offices for staff, four in number (208, 212, 214 and 216), two of which are fitted out as laboratories; and a small lecture theatre (112).

The course leading to the degree of sivilingeniör in metallurgy involves two years study of the basic subjects: mathematics, physics, chemistry, geology, mineralogy, crystallography, mechanics, mechanical technology and theory of machines, followed by two senior years devoted to the special subjects: electrotechnics, petrography, geology, ore geology, physical chemistry, machine design, physical metallurgy, social economy, ore dressing and process metallurgy with emphasis on thermodynamics, kinetics and unit operations.

Visits to local works are made at intervals and in the early summer, works excursions of two to three weeks'

duration are arranged for senior students, often in other countries as well as Norway.

All metallurgical students are required to have spent at least eleven months in practical work before commencing their course. Of this period five months must be on a metallurgical plant and the remainder in an ordinary engineering workshop. Many students take advantage of the existing international exchange schemes to visit other countries for at least part of their practice. In addition to these requirements and to the final examinations all graduating students spend three to four months of their final year on a set problem on which they submit a report.

There is at present extremely keen competition for admission to the Technical University in all departments and graduates as elsewhere in the world are often engaged by industry before they have completed their courses.

The foregoing has covered the facilities for technical education at a university level in metallurgy now provided in Norway. This account would, however, be incomplete without some reference to the lighter side of student life in Trondheim. There is general agreement that Trondheim is the student town par excellence. The students union, which comprises all matriculated persons of all ages in the neighbourhood, provides a forum for discussion of all problems of topical interest and strives to secure guest speakers of international magnitude. Last autumn the president, who was incidentally a metallurgical student, secured Bertrand Russell for the Jubilee meeting and had a promise of Professor Reuther, Burgemeister of beleaguered Berlin, for a later meeting. A colleague of Reuther's had to deputise for him when the time came unfortunately, but the bold experiment of inviting a German to address an audience in a country that was occupied by Germany only three years previously was otherwise highly successful. Students in the various departments of the Technical University have their own societies or fraternities with emphasis on conviviality with a modicum of shop. The University Athletic Union has a fine alpine hut on the mountain side, about 8 km. from the town, with catering and accommodation for a number of residents.

A Note on the Accuracy of Analysis

By E. Scheuer* and F. H. Smith†

With a view to stimulating discussion on the accuracy of analytical methods, the authors present the results of the examination of a large number of analyses of aluminium alloys carried out in several laboratories.

IN dealing with analysis methods, it is often found desirable to have a yardstick with which to compare the accuracy obtained by a certain method in order to find out if it can be used for normal production work. The obvious way is, of course, to compare the analytical accuracy with the composition limits of the specification prescribed for the production. Unfortunately not all specifications are sufficiently realistic to serve as a guide to the judgment of the analyst, as many of them ask for

an accuracy which it is not possible to achieve in practice. This is due either to lack of appreciation of analytic limitations by the persons who lay down the specifications, or to metallurgical requirements which have not yet been supplemented by suitable analytical means of routine control.

When the Chemists Committee of ALAR (Association of Light Alloy Refiners) was confronted with the problem of defining, in general terms, a practical and useful accuracy for the *routine analysis*‡ of aluminium alloys, one of the authors suggested studying the accuracy of analysis methods in relation to contents on a statistical basis. A considerable volume of evidence was

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† Development Officer, ALAR, Ltd.

‡ It is clear that with highly-skilled workers and the necessary time, higher degrees of accuracy can be attained.

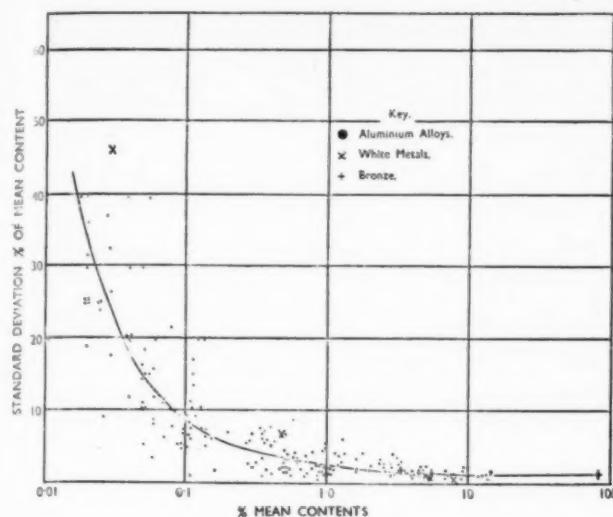


Fig. 1.

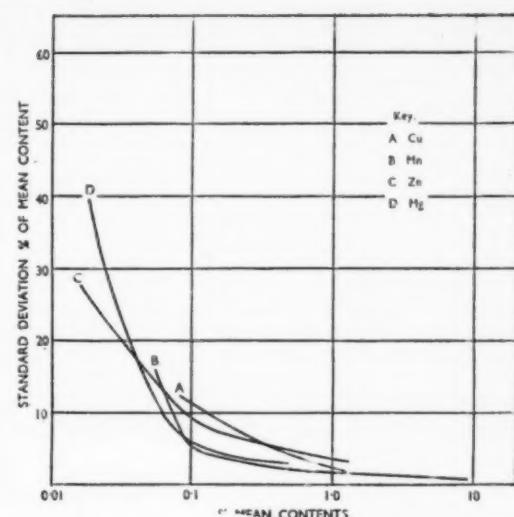


Fig. 2.

available in the form of analysis results on exchange samples analysed by various laboratories of member firms and also from standard samples analysed many times in the same laboratory under routine conditions. A graph was prepared by plotting the coefficient of variation (standard deviation in % of mean content) of analysis results obtained on specific samples of aluminium alloys against the average content, irrespective of the element (Fig. 1).

This is obviously a very rough method, but, in spite of this, it yielded a graph which showed a comparatively clear relation between the amount of element present and the coefficient of variation. This is confirmed by graphs which represent the same relation for single elements, as shown in Fig. 2 for copper, manganese, zinc and magnesium, and which are very similar to each other.

It might be noted here that the results embodied in this graph are obtained using a variety of routine methods: volumetric, gravimetric, colormetric and

polarographic—often two or three different methods for the same element.

It was further thought to be of interest to find out how the position in the analysis of other metals compares with that in the field of aluminium. For this purpose a few figures from the routine analysis of bronze and white metals were made available. The values for these results are entered in Fig. 1, and fit in quite well with the graph for aluminium alloys.

A further graph on the same lines was produced from the results of spectrographic analysis of aluminium alloys. The number of results available for the construction of this graph was much smaller than for chemical analysis, and the graph (Fig. 3, Curve C) should be considered only as a first approximation and subject to confirmation. It is based on the deviations found by each laboratory from their repeated analysis of standard samples. Curve B is a similar curve for chemical analysis. (In curve A the deviations are calculated from single results, submitted by the six laboratories, on each of a number of samples.)

It appears that the spectrographic results are likely to be more accurate than chemical results for contents below about 0.1%, whilst chemical methods are more accurate above about 0.1%. The exact position of the point of intersection of graphs B and C is of course very uncertain; most spectrographers will probably feel that the graph does not in fact represent the true relationship between chemical and spectrographic methods, and is too unfavourable to the spectrograph.

It is not easy to explain exactly the significance of this evidence. The general trend of the coefficient of variation to increase with decreasing contents is easily understood. The authors feel, however, that it is remarkable how little the graphs for single elements (Fig. 2) deviate from each other and from the general graph, and also how well the figures for tin alloys and bronze fit into the general graph. This seems to indicate more than just a common general trend.

If we consider that the selection of routine analysis methods is a compromise between accuracy requirements for ideal technical control and economy in skill and time required, the results of this investigation

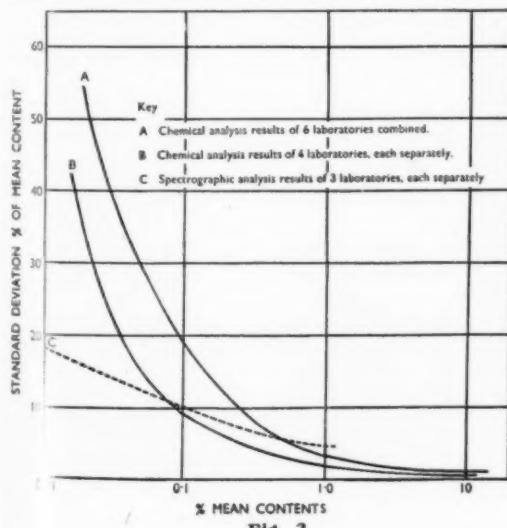


Fig. 3.

would indicate that the level of accuracy at which this compromise is achieved is mainly dependent on the mean content of the element to be determined and much less on the nature of the element or even on the alloy to be analysed.

It is understood that the Aeronautical Inspection Directorate have for some years used a formula

$$\text{Tolerance} = \pm \sqrt{K_1 c + K_2}$$

in which c is concentration and K_1 and K_2 are constants, in assessing the degree of acceptable correspondence between two sets of results. If the A.I.D. tolerances are now considered as three times the standard variation

limits, the agreement with the author's results is remarkably close.

It has been suggested that by plotting standard deviation against the content the graph assumes a simpler shape, as is evident from the A.I.D. equation, and the data might even fit a straight line equation. The authors would, for the moment, prefer not to enter into theoretical discussions. Their aim is to stimulate publication of similar evidence from other laboratories in order to check the significance of their observations.

The ALAR Chemists' Committee, on whose behalf this note is presented, are carrying out further checks on the usefulness of Graph A by comparing with it the deviations of their periodical exchange analysis samples.

The Finch Electron Diffraction Camera

The practical application of electron diffraction is becoming increasingly appreciated ; it has been proved to be a powerful research technique for the investigation of the structure of matter, and particularly for the study of thin films, surfaces and the structure of gases. This technique is applied by means of the electron diffraction camera and in this article the Finch camera is described.

ELECTRON diffraction is being employed to an increasing extent, particularly for the investigation of the structure of thin films and surface layers of metals, metallic oxides and compounds generally, oils, laquers, elastomers, fibres, etc. Its practical application has already met with considerable success in investigating surface phenomena connected with, for example, corrosion, metal to rubber bonding, plating, lubrication, catalytic activities of surface films, poisoning of oxide coated filaments, etc. In view of the progress made in this field it is of interest to give a brief description of an instrument developed for the study of these phenomena by means of electron diffraction

Fundamentals of the Electron Diffraction Camera

In principle the camera consists of :—

1. A device for producing a fine beam of electrons moving at a controlled velocity. Electrons accelerated through a difference of potential of 50 kv. have a wave length of about 5×10^{-8} cm. which is of the order required for the examination of crystal lattices. The practical applications of electron diffraction which have been employed up to date find the voltage range 40–65 kv. to be the most useful.
2. An arrangement for holding the specimen in any desired position relative to the beam.
3. Visual and photographic means for observing and recording the angular distribution of the diffracted electrons.

From Bragg's Law relating the diameter of any given diffraction ring (a polycrystalline specimen for example) to the lattice spacing, it can be shown that the diameter of the ring depends also on the wave length. In consequence, any change in accelerating voltage during exposure will lead to a broadening of the diffraction rings or possibly doubling of the pattern. For the best definition it is, therefore, imperative for the accelerating voltage to have a high order of stability ; the high tension system has to be designed accordingly.

Electron Beam Production.—The methods of stabilisation depend primarily on the type of source used for

the electron beam. In the hot cathode system, stabilisation of the accelerating voltage is usually obtained by the use of complex electronic circuits.

The Edwards diffraction camera, which is described in this article and illustrated in Fig. 1, uses the cold cathode discharge as a source of electrons together with the Finch H.T. circuit because of the excellent performance, inherent simplicity and reliability, which can be obtained with properly designed apparatus of this type. The cold cathode discharge gives an electron beam of high current density and can be used for accelerating voltages of 15–80 kv. The most useful range of accelerating voltages for structure analysis lies between

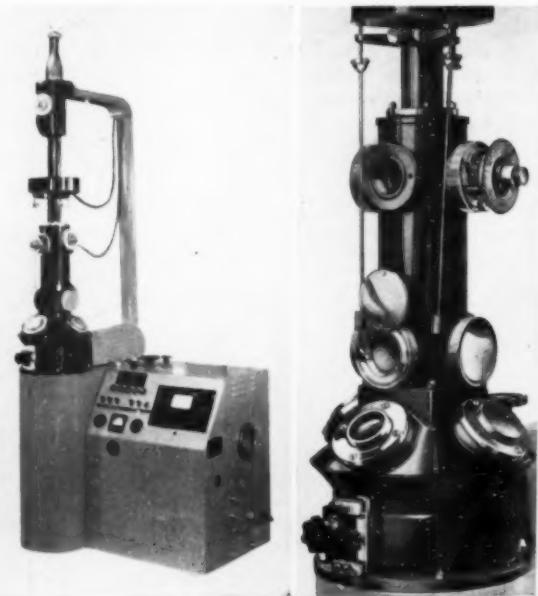


Fig. 1.—The Finch Electron Diffraction Camera.

Fig. 2.—Close up view of specimen and camera sections.

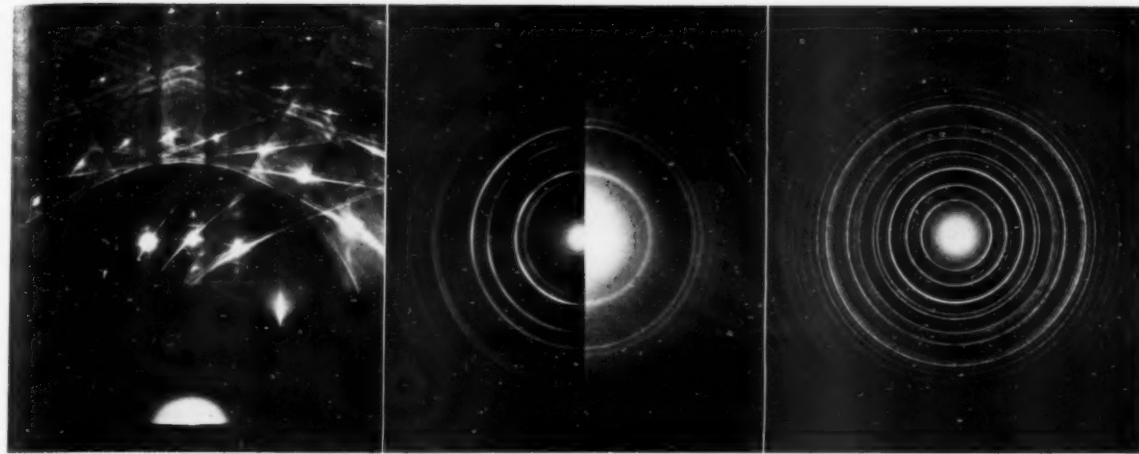


Fig. 3.—Typical diffraction patterns taken with the Finch camera : (a) Single crystal of silicon carbide ; (b) Split shutter comparison exposure of gold (left) and graphite (right) for calibration against standard ; (c) Partly oxidised tin foil showing typical transmission pattern of a polycrystalline substance. These illustrations of the diffraction patterns were reproduced from the original plates. Standard size, $3\frac{1}{4} \times 4\frac{1}{2}$ ins. (Loss of detail is unavoidable during reproduction).

30 and 70 kv. and under these conditions a cold cathode source is robust and simple, a monochromatic electron beam being easily obtained.

The discharge conditions in the cold cathode tube are interdependent, i.e., the gas pressure, current passing in the discharge and accelerating voltage are related, so that any one is a function of the other two factors. Use is made of this property in the stabilisation. The gas pressure is kept constant by a special leak system and a circuit employing the saturation characteristics of a diode ensures a constant current, so that constant acceleration voltage is obtained with relatively simple equipment.

Diffraction Techniques.—There are three principal methods of electron diffraction :—

- (a) Transmission—in which exceedingly thin films of the specimen material are prepared from foils, by evaporation *in vacuo*, or by cathodic sputtering, etc.
- (b) Reflection—in which the electron beam falls upon the specimen at grazing incidence, for the examination of specimens such as surface layers of massive material or very thin films.
- (c) The examination of substances in the gaseous or vaporous state, providing an extremely useful method of analysis.

Methods of Measuring.—In the interpretation of the diffraction patterns, the lattice parameters may be calculated within 2% from the constants of the apparatus, and the value of the accelerating voltage. This accuracy is dependent only on the measurement of the accelerating voltage. A salient feature of this instrument, however, is the fact that it is not necessary to attempt any absolute measurements involving these various constants, as diffraction patterns obtained from specimens under investigation may be referred to comparison patterns obtained from standard substances of which the lattice spacings are already known, thus giving to electron diffraction techniques the accuracy of measurement of X-ray diffraction. To facilitate this reference technique, a special split plate shutter has been designed so that separate exposures can be obtained in two independent halves, one from the specimen and one from the reference substance. The specimen carrier

permits either substance to replace the other in the electron beam by simple manipulation.

The Finch Camera made by W. Edwards

The electron diffraction camera and H.T. unit for the operation of the cold cathode source are based on the well-known form and principles developed by Professor G. I. Finch. The camera equipment to be described embodies modern vacuum engineering design, the various demountable joints being quickly and easily made, which joints, together with the operating movements, are effectively sealed with elastomer O-rings, metal bellows and Wilson shaft seals, thus dispensing with the grease-sealed mating surfaces hitherto employed.

The camera body is designed integrally with the supporting cabinet so that all services can be adequately and conveniently provided as a complete unit ; a compartment extending from the cabinet and running vertically behind the camera body houses the cathode leakage cooling water and electrical services to the upper camera body. The electrical instruments and controls are fitted to the sloping panel of the aluminium cabinet. The camera body and component parts are fabricated from non-magnetic material either solid or drawn or cast specially to ensure freedom from porosity.

The camera consists fundamentally of four principal sections :—

- (a) Cathode chamber assembly.
- (b) Collimating and focusing section.
- (c) Specimen section.
- (d) Camera chamber.

These sections are readily dismantled for cleaning purposes or for facilitating special alterations to suit particular work, with the aid of convenient high vacuum seals. A close-up view of the specimen and camera sections is shown in Fig. 2, and some typical diffraction patterns taken with this camera are reproduced in Fig. 3.

Cathode Chamber Assembly.—A glass cathode chamber is secured with a demountable vacuum joint and is adjustable with respect to the water-cooled anode block. The aluminium cathode is removable from the top of the glass chamber, so that repolishing of the cathode can be carried out as required.

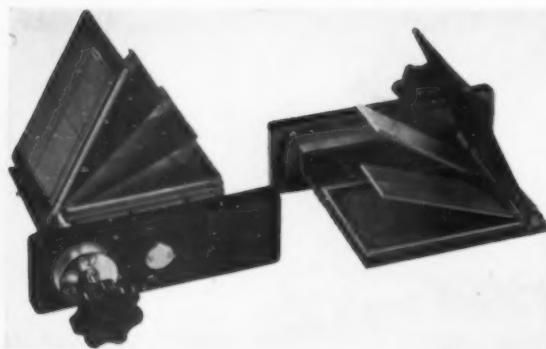


Fig. 4.—Plate holders : (left) Triple plate holder ; (right) Split Shutter single plate type.

The discharge pressure is maintained at an easily adjusted constant value by a separate small oil diffusion pump and a fixed leak from a "leak chamber." The leakage rate is controlled by the leak chamber pressure which can be accurately adjusted by a fine control needle valve to raise the pressure, and a special valve and connection to an evacuated reservoir chamber to decrease the pressure as required. The latter adjustment can be carried out without interfering with the pumping of the camera, i.e., while the H.T. is on. A small manometer indicates the leak chamber pressure and a Pirani gauge in the cathode chamber pumping line indicates the discharge pressure. A steady and easily controllable discharge is obtained by the use of this arrangement. Provision is also made for using other gases in the discharge—to prevent, for example, oxidation of a delicate specimen.

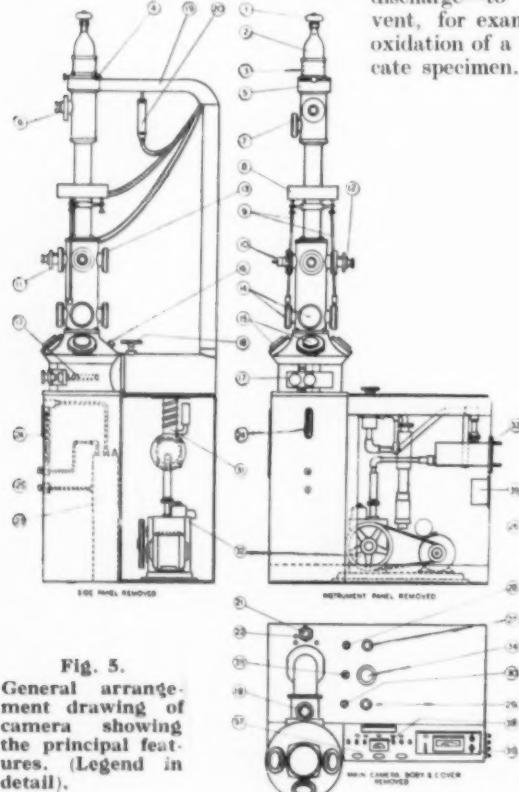


Fig. 5.
General arrangement drawing of camera showing the principal features. (Legend in detail).

Collimating and Focusing Section.—The beam is collimated by a fixed diaphragm in a water-cooled anode block and an aligning diaphragm, followed by magnetic concentration. The adjustable diaphragm is held in a demountable carrier which permits reciprocal and rotary motion. In this way the beam containing high speed electrons, molecular rays and electrons which have lost energy by elastic collisions, can be inclined to the main axis of the apparatus and thus, with the concentrating field suitably adjusted and biased, the molecular rays remain trapped, but the electrons, both high and low speed, are released to pass into the specimen chamber in the form of a magnetic spectrum, the undesirable electrons being eliminated in the specimen section, as described below. An inspection port is provided adjacent to the diaphragm port.

The focusing coil can be tilted with adjusting rods easily operable whilst viewing, to move the electron beam for the purpose already described, to direct it at any part of the specimen or to explore it ; the beam is focused on or just below the fluorescent screen by adjusting the field strength of the focusing coil. The focusing coil current is derived from a stabilised mains-operated power unit.

Specimen Section.—Specimens can be mounted at two different levels, giving a diffracted beam length of 50 and 25 cm. respectively. At each level four ports are provided, each of which is designed to carry :—

- (a) A specimen carrier with movements permitting inclination of the specimen plane to electron beam, translation into beam, rotation in the azimuthal plane, lateral motion of the specimen plane at right angles to the electron beam.
- (b) A diaphragm carrier with movements similar to the specimen carrier and carried immediately above the specimen for the filtration of the slower and unwanted electrons from the electron beam.
- (c) A decharger (consisting, essentially, of a low-voltage electron gun for which the filament current

DISCHARGE CHAMBER.

- 1. Cathode holder and aluminium cathode.
- 2. Glass discharge chamber.
- 3. X-ray protection shield.
- 4. Adjustment for discharge chamber alignment.
- 5. Water-cooled anode block containing anode diaphragm.

COLLIMATING AND FOCUSING SECTION.

- 6. Collimating diaphragm holder.
- 7. Inspection port.
- 8. Magnetic focusing and scanning lens.
- 9. Scanning adjustment.

SPECIMEN SECTION.

- 10. Decharger spray gun (low power electron gun).
- 11. Diaphragm holder.
- 12. Specimen holder.
- 13. Inspection port.
- 14. Lower four ports at 25 cm. diffracted beam lengths.

CAMERA SECTION.

- 15. Inspection window for visual observation.
- 16. Air release valve.
- 17. Photographic plate cassette.

VACUUM EQUIPMENT AND COMPONENTS.

- 18. Tilting plate isolation valve.
- 19. Cover for pumping, leak and water lines.
- 20. Pirani Gauge Head.
- 21. Discharge chamber pumping line.
- 22. Fixed leak to discharge chamber.
- 23. Leak reservoir.
- 24. Manometer for reservoir pressure.
- 25. Isolation valve between leak line and reservoir.
- 26. Discharge chamber diffusion pump.
- 27. Discharge chamber isolation valve.
- 28. Discharge chamber roughing valve.
- 29. Reservoir pumping valve.
- 30. Needle Valve to increase reservoir pressure.
- 31. Diffusion Pump for camera (60 lit./sec.).
- 32. Rotary Pump (displacement 150 cu. ft./min.).
- 33. P₀ moisture trap.
- 34. Rotary pump selector valve.
- 35. Air release to rotary pump.
- 36. Pirani gauge, for leak testing and recording of backing and discharge pressures.
- 37. Instrument panel for lens supply decharger, etc.
- 38. Focusing lens coil supply meter.
- 39. Water flow switch giving protection to diffusion pump in the event of water failure.

and accelerating voltage is supplied from a suitable power unit) for neutralising the charge acquired by non-conducting specimens, which otherwise result in distortion of the diffraction pattern.

(d) Inspection port. These ports are all interchangeable in position as required. Port blanks are provided which may be adjusted for specialised work such as fitting insulators to carry filaments for the evaporation of metals and salts, or thermocouples, etc., into the specimen section according to the special requirements of the individual research worker. Specimens are mounted in interchangeable holders for the transmission and reflection techniques.

Camera Chamber.—The plate holder is quickly demountable from the camera body, a rubber seal and quick-action securing bar being employed. The plate holders at present available are of three types:—single plate holder for taking one exposure only; triple plate holder for exposing up to three plates without breaking the vacuum; and the split plate shutter, Fig. 4, which permits the two halves of a plate to be exposed independently for comparison purposes, each type having a fluorescent screen upon which the preliminary focusing is carried out. A holder to take roll film will shortly be available. Three inspection windows are provided for

observation of the fluorescent screen. A drawing showing the general arrangement of this camera and indicating the principal features is given in Fig. 5.

High Tension Supply.—The constant current high tension supply system for the operation of the cold cathode tube follows Professor Finch's basic specification for the saturated diode circuit which has been so successfully employed by him.

A rectified high voltage supply ($\frac{1}{2}$ wave) is applied to a high voltage condenser. Current flows to the cathode of the decharger chamber through a diode, run under conditions of saturation; the filament temperature of the diode controlling the high tension current.

H.T. equipment is available basically according to Professor Finch's specification and physically to suit the camera arrangements. The equipment has been designed for compactness and ease of operation and consists essentially of two parts; the generator, comprising high tension and valve filament transformers, rectifier unit and condenser all oil-immersed in a single tank, and a control table of the portable desk type, incorporating mains switch, kv. control and indicator, and a spark gap for measuring the accelerating voltage.

We are indebted to W. Edwards & Company (London) Limited for permission to present this article and for the use of photographs from which the illustrations were reproduced.

Metallo-organic Reagents used in Microanalysis—II

By F. Lester

Continuing the review of suitable weighing forms for various metals in micro gravimetric analysis, methods are now described for the determination of mercury, silver, antimony, manganese, cobalt, nickel, silicon, copper and zinc.

Determination of Mercury

Precipitation with ethylenediamine.—Mercury is quantitatively precipitated as $(\text{Cuen}_2)(\text{HgI}_4)$ when an excess of copper ethylenediamine nitrate $(\text{Cu en}_2)(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$ is added to a neutral or slightly ammoniacal solution containing mercury and excess potassium iodide.¹

The solution, which should occupy a volume of 40–100 ml., is treated with an excess of potassium iodide and heated to boiling. A boiling solution of the reagent is then added in excess and the mixture stirred well. After cooling, the precipitate is filtered on a filter-crucible, the transference being effected by a 0.1% solution of potassium iodide containing 0.1% of the precipitant. The precipitate is washed several times with the same solution, then 3–4 times with 2 ml. portions of alcohol and finally 2–4 times with 2 ml. portions of ether. After drying for 15 mins. *in vacuo* the precipitate is weighed. The factor for mercury is 0.2249.

Mercury may also be determined titrimetrically by titrating with the reagent until a violet colour appears. The end-point may also be determined potentiometrically.

Precipitation with propylenediamine.—Mercury may be determined similarly by precipitation with copper propylenediamine sulphate.⁴ The method is similar in detail to that preceding. The factor for mercury is 0.2181. Mercury may be precipitated in the presence of copper, cobalt, chromium, nickel and zinc.

Precipitation with benzidine and tolidine.—When a solution of mercury salts containing a halide or cyanide is treated with a solution of benzidine in 50% acetic acid complexes of the formulae $(\text{HgBzd})\text{Cl}_2$, $(\text{HgBzd})\text{Br}_2$, $(\text{HgBzd})\text{I}_2$ and $(\text{HgBzd})(\text{CN})_2$ are precipitated. The compounds are sufficiently insoluble to be used as weighing forms for the determination of mercury. The precipitates are washed with alcohol and ether and dried *in vacuo* prior to being weighed.

O-tolidine precipitates similar complexes which may also be used for the determination of mercury.⁶

Precipitation with α -naphthylamine.—Mercury is precipitated quantitatively as $\text{Hg}(\text{C}_{10}\text{H}_7\text{NH}_2)_2(\text{CNS})_2$ when an alcoholic solution of α -naphthylamine is added to a solution containing a mercuric salt and thiocyanate ions.⁷ The method does not appear to have any particu-

¹ Spacu, G., and Spacu, P. *Ibid.*, 1932, **59**, 187.

² Spacu, G., and Armeanu, V. *Bull. Soc. Stiinte Cluj.*, 1934, **7**, 621.

³ Spacu, G., and Murgulescu, I. G. *Z. Anal. Chem.*, 1934, **96**, 109.

⁴ Spacu, G., and Spacu, P. *Ibid.*, 1932, **59**, 187.

⁵ Mandal, E. L. *Science and Culture*, 1940, **5**, 719.

⁶ Herzog, W. *Chem. Ztg.*, 1926, **50**, 642.

⁷ Ubeda, F. B., and Alloza, R. *As. Fis. quim.*, 1941, **37**, 350.

lar merit since the precipitate dissolves in excess of potassium thiocyanate solution and accordingly, the amount of the latter must be carefully controlled.

Precipitation with pyridine.—When pyridine is added to a solution containing a mercury salt and ammonium dichromate, an orange-yellow precipitate of (HgPy_2) Cr_2O_7 is formed. The precipitate is sufficiently insoluble to be used as a weighing form for mercury.⁸

About 2 g. of ammonium dichromate are added to the solution containing the mercury salt and the mixture is diluted to 100 ml. When the crystals have dissolved completely, 1 ml. of pyridine is added immediately and the mixture is well stirred. After about 5–10 mins. the precipitate is filtered through a Gooch crucible and washed 4–5 times with a wash solution containing 0.5 g. of ammonium dichromate and 0.5 ml. of pyridine in a litre of water. Washing is then continued with 80% alcohol containing 1 drop of pyridine for each 10 ml. of alcohol used in preparing the 80% alcohol. Finally, the precipitate is washed with ether, dried in *vacuo* and weighed. The factor for mercury is 0.3490.

Furman and State⁹ have simplified the washing procedure by substituting methyl acetate for the various wash liquids used. They also used volumetric methods to complete the determination by dissolving the precipitate in acid and titrating either iodometrically or ferrous-metrically. Copper, cadmium, cobalt, nickel, zinc, manganese, chromium, lead, silver and iron interfere and must be removed before the determination of mercury.

Determination of Silver

Precipitation with ethylenediamine.—Silver may be determined quantitatively by precipitation with cobalt ethylenediamine dithiocyanato-thiocyanate in the presence of potassium thiocyanate.¹⁰ The precipitate consists of $(\text{Ag}(\text{CNS})_2)(\text{Co en}_2(\text{CNS})_2)$. The cobalt reagent ($\text{Co en}_2(\text{CNS})_2\text{CNS}$) is prepared by dissolving 80 g. of cobalt chloride crystals in 200 ml. of water and adding 60 ml. of ethylenediamine dissolved in 40 ml. of water. Air is drawn through the brown-red liquid for 12 hrs. and then 400 ml. of concentrated hydrochloric acid are added. The mixture is heated for several hours until a clear blue liquid is obtained which is allowed to stand for several days. The crystals which separate are filtered, washed with concentrated hydrochloric acid, then with alcohol-ether solution (1 : 1) and finally with ether. The yellowish-green crystals of cobalt ethylenediamine chloride are dried over sulphuric acid for 24 hrs. Forty grams of the compound are dissolved with warming in 60 ml. of water and mixed with a warm solution of 40 g. of potassium thiocyanate in 30 ml. of water. A light green precipitate forms as a paste. The mixture is boiled and the precipitate goes through several colour changes to a nearly black colour. When cooled the mass solidifies to a paste. On further boiling the mass turns red and when cool gives a mass of red-brown crystals. These are diluted with 5–6 volumes of water, boiled and filtered. The product is crystallised from the filtrate and recrystallised from water several times. An aqueous solution of this material is mixed with potassium thiocyanate and heated to boiling. When it is cooled, a blood-red crystalline mass of the reagent separates. The filtrate is treated with more potassium thiocyanate and evaporated to recover more of the reagent. The reagent is finally purified by crystallising several times from water.

The solution of the silver salt should be neutral or

only faintly acid to nitric acid. Potassium thiocyanate is added until the precipitate of silver thiocyanate just redissolves. The solution is heated to boiling and a concentrated solution of the reagent is added. When cool, the reddish-orange precipitate is filtered and washed with a 1% potassium thiocyanate solution containing 0.3–0.5% of the reagent. Washing is continued with alcohol and ether. The precipitate is then dried in *vacuo* and weighed. The factor for silver is 0.2078.

Precipitation with propylenediamine.—Silver may also be precipitated with copper propylenediamine sulphate in the presence of potassium iodide.¹¹ The precipitate has the formula $(\text{AgI}_2)_2(\text{Cu pn}_2)$. The procedure is similar to the one described previously. The factor for silver is 0.2807.

Determination of Antimony

Precipitation by ethylenediamine.—When chromium ethylenediamine chloride ($\text{Cr en}_3\text{Cl}_3 \cdot 3.5\text{H}_2\text{O}$) is added to a solution containing sodium thioantimonate Na_3SbS_4 the compound $(\text{Cr en}_3\text{SbS}_4 \cdot 2\text{H}_2\text{O})$ is precipitated. It is extremely insoluble in water and is very stable. The yellow precipitate is so voluminous that the method is limited to a maximum of 50 mg. of antimony.¹²

The reagent is prepared by mixing 8 g. of chrome alum, previously dried at 100°C. with 6 g. of ethylenediamine in a flask fitted with an air condenser. The mixture is heated on the water bath and after a few hours a yellow to red mass forms. This is mixed into paste with water and the yellow salt dissolves leaving the insoluble red $(\text{Cr}_4(\text{OH})_6\text{en}_6)(\text{SO}_4)_3$ behind. The mixture is filtered and the yellow solution mixed with ammonium chloride to precipitate the reagent as a yellow solid. This is recrystallised twice from hot water.

Ammonia is added to the antimony solution until a faint odour of ammonia persists. The solution is heated to boiling and about 0.5–1.0 g. of pure sodium sulphide is added. If the precipitate does not dissolve, more sulphide is added. A few drops of sodium polysulphide are added to clear the solution and the heating continued for a short time. The solution is diluted for 50 mg. Sb. to 300 ml., heated to 70°–80°C. and a threefold excess of cold reagent solution added. When cool the beaker is placed in cold water for 2 hrs. and then the precipitate is filtered on a Gooch crucible. It is washed with water, then with alcohol and finally with ether and dried over calcium chloride in a desiccator for 15 mins. The factor for antimony is 0.23494.

Determination of Manganese

Precipitation with pyridine.—Manganese may be determined rapidly and accurately by precipitation with pyridine in the presence of thiocyanate ions.¹³ The precipitate consists of $(\text{MnPy}_2)(\text{CNS})_2$. The determination is conducted similarly to the other methods employing pyridine as the precipitant, and organic wash liquids are used. Many other ions interfere. The factor for manganese is 0.1127. The method appears to be of little practical value since manganese can readily be determined in the presence of many other metals by conventional procedures.

Determination of Cobalt and Nickel

Precipitation with Pyridine.—Cobalt is precipitated as the complex $(\text{CoPy}_4)(\text{CNS})_2$ by pyridine and thio-

⁸ Spacu, G., and Dick, J. *Z. anal. Chem.*, 1929, **76**, 273.

⁹ Furman, N. H., and State, H. M. *Ind. Eng. Chem. (Anal.)*, 1936, **8**, 467.

¹⁰ Spacu, P. *Bull. Soc. Stiinte Cluj*, 1934, **7**, 568.

¹¹ Spacu, G., and Spacu, P. *Z. anal. Chem.*, 1932, **90**, 182.

¹² Spacu, G., and Pop, A. *Ibid.*, 1938, **111**, 254.

¹³ Spacu, G., and Dick, J. *Ibid.*, 1928, **74**, 188.

cyanate.¹⁴ Copper, cadmium, nickel, manganese and zinc interfere. The precipitation is effected as in the other methods employing pyridine as the precipitant. The factor for cobalt is 0.12002. The method has been applied to the separation of cobalt in steels. Nickel forms an analogous compound and may be determined in the same manner.¹⁵ In some cases it is ignited to the oxide, in which case there is no advantage to be gained in using the method.

When nickel and cobalt are present in the same sample they can be precipitated as the complex thiocyanates and weighed.¹⁶ Nickel is then determined in another portion of the solution by the dimethylglyoxime method and the cobalt obtained by difference.

Both metals may be determined volumetrically by adding to their pyridine solution, excess standard thiocyanate, filtering off the complex, and determining the excess thiocyanate by titration with silver nitrate.¹⁷

Dobbins and Sanders¹⁸ found this method unsatisfactory. They state it is impossible to wash out all the excess thiocyanate without dissolving some of the complex. By modifying the method, satisfactory results were obtained. The sample (50–100 mg. Co) is transferred to a 250 ml. graduated flask, dissolved in 150 ml. of water, made just acid to litmus with nitric acid and then 3 ml. of pyridine added. An excess of 0.1 N ammonium thiocyanate is added, the contents of the flask are diluted to the mark well mixed and filtered. The first few ml. of the filtrate are discarded. Fifty ml. of the filtrate are transferred to a heater, diluted to 100 ml. and the thiocyanate determined by the Volhard method. This procedure is claimed to give excellent results.

Nickel may also be determined indirectly by precipitation of the pyridine thiocyanate complex using standard thiocyanate, and determining the excess of the latter potentiometrically by titration with standard silver nitrate solution.¹⁹

Determination of Silicon

Precipitation with pyridine.—Pyridine precipitates silicomolybdate acid quantitatively from an acid solution containing a silicate and excess of molybdate. The complex has the formula $(C_{13}H_{17}N_3O) \cdot H_8Si(Mo_2O_7)_6 \cdot 6H_2O$. It is washed with ice-cold 0.1 N hydrochloric acid and dried at 60°–70° C. for 45 mins. prior to being weighed.²⁰

Precipitation with pyridine.—Silicomolybdate acid forms a similar complex with pyridine, but it is not very satisfactory as a weighing form for silicon. It can, however, be titrated either by reduction of the molybdenum and titration with permanganate²¹ or acidimetrically.²²

Mika²² has developed a method for the micro-determination of silicon in steel using this process. 0.1 g. of steel is dissolved in dilute nitric acid, and ammonium molybdate is added to the hot solution. The solution is maintained at 90° C. for 5 mins. Pyridine, acidified with nitric acid is added in excess, the solution is cooled, filtered and the precipitate washed with potassium nitrate solution. The washed precipitate is dissolved in a measured excess of 0.1 N sodium hydroxide

solution and the excess back-titrated using phenol red as indicator. 1 ml. 0.1 N NaOH = 0.1169 mg. Si.

Precipitation with 8-hydroxyquinoline.—8-Hydroxy-quinoline precipitates a complex silicomolybdate from acid solution. The silicon content is determined indirectly by bromination of the 8-hydroxyquinoline portion of the complex.²³ The method has been applied to the determination of silica in minerals²⁴ and in slags.²⁵

Determination of Copper

Precipitation with ethylenediamine.—In a neutral or slightly acid solution, copper in the presence of potassium mercury iodide K_2HgI_4 is precipitated by ethylenediamine. The precipitate consists of $(Cu_{en})_2HgI_4$.²⁶ It is weighed after washing and drying. The factor for copper is 0.07126.

Precipitation with pyridine.—Pyridine precipitates copper in the presence of thiocyanate as the complex $(CuPy_2) (CNS)_2$.²⁷ The precipitate is washed with special wash solutions and finally with alcohol followed by ether. After drying *in vacuo* the precipitate is weighed. The factor for copper is 0.18817.

By slight modification to the method copper can be separated from mercury, zinc, lead and antimony.

*Precipitation with *a*-naphthylamine.*—Copper is precipitated as a complex thiocyanate when *a*-naphthylamine is added to a solution containing these ions.²⁸ The reaction is sensitive to 1 : 300,000. The precipitate consists of $Cu (C_{10}H_7NH_2)_2 (CNS)_2$ and may be used as a weighing form in the determination of copper.

Other methods.—Copper may be determined by precipitation with propylenediamine in place of ethylenediamine.²⁹ The procedure is identical. Complex thiocyanates of copper are precipitated by benzidine and *o*-tolidine,³⁰ but the methods have no practical value as the precipitates have to be ignited to the oxide.

Determination of Zinc

Precipitation by pyridine.—When a solution containing zinc and a slight excess of ammonium thiocyanate is treated with a few drops of pyridine a white precipitate of $(ZnPy_2) (CH_3)_2$ is formed. A large excess of pyridine redissolves the complex and must be avoided. The precipitate is treated as in other methods where pyridine is used as precipitant, being washed with special wash solutions and organic solvents. It is finally weighed as $(ZnPy_2) (CNS)_2$, the factor being 0.1925.³¹

The precipitate may also be titrated.³² If iodine is added to the slightly alkaline solution the following reactions occur:



The excess iodine is determined by titration with sodium thiosulphate.

Precipitation by tetraphenyl arsonium chloride.—In highly concentrated solutions of chloride containing zinc ions, the above reagent precipitates the complex $(C_6H_5)_4As \cdot ZnCl_4$. The chloride solution must be concentrated since the compound is soluble in water. A standard solution of the reagent is used³³, and after filtration of the precipitate, the excess is determined iodimetrically.

¹ Spacu, G., and Dick, J., *Ibid.*, 1927, **71**, 97.

² Spacu, G., and Dick, J., *Ibid.*, 1927, **71**, 442.

³ Spacu, G., and Macarović, C. G., *Ibid.*, 1936, **8**, 444.

⁴ Spacu, G., *Bull. Soc. Sci. Chim.*, 1922, **1**, 314.

⁵ Dobbins, J. T., and Sanders, J. P., *Ind. Eng. Chem. (Anal.)*, 1934, **6**, 459.

⁶ Spacu, G., *Z. Anal. Chem.*, 1934, **96**, 245.

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¹⁰ Berg, R., and Teitelbaum, M., *Z. Angew. Chem.*, 1928, **41**, 611.

¹¹ Vonunetz, M. I., and Bernstein, S. S., *Zar. Lab.*, 1936, **5**, 1,071.

¹² Ginsburg, L. B., *Ibid.*, 1938, **7**, 1,041.

¹³ Spacu, G., and Suciu, G., *Ibid.*, 1929, **78**, 329.

¹⁴ Spacu, G., and Dick, J., *Bull. Soc. Sci. Chim.*, 1928, **4**, 23.

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¹⁶ Spacu, G., and Spacu, P., *Z. anal. Chem.*, 1932, **89**, 187.

¹⁷ Spacu, G., and Macarović, C. G., *Ibid.*, 1935, **102**, 350.

¹⁸ Miller, C. F., *Chemist Analyst*, 1931, **29**, 8.

¹⁹ Pagel, H. A., and Ames, O. C., *J.A.C.S.*, 1930, **52**, 3093.

²⁰ Willard, H. H., and Smith, G. M., *Ind. Eng. Chem. (Anal.)*, 1939, **11**, 269.

Modern Methods of Gas Analysis

The G.L.C. Constant Volume Apparatus

By W. D. Vint

Since the publication of a series of articles on the above subject, the eighth part of which appeared in the April, 1948 issue, the author has considered the inclusion of the G.L.C. constant volume apparatus, the principle and application of which is described in this article.

THE principle of the apparatus is based mainly, on that of the Bone and Wheeler gas analyser, and this depends upon the fact that providing the volume of a gaseous mixture is kept constant, the difference in pressure occasioned by the removal of one of the gaseous constituents is proportional to the volume of that constituent. The G.L.C. apparatus can, however, claim to possess several advantages over the original Bone and Wheeler instrument and a number of modifications with a view to correcting the errors incurred by most other methods of gas analysis have been made.

The particular advantages claimed are as follows:—

1. No absorption errors in the combustion tubes as the carbon dioxide absorbed by the copper oxide is released when the latter is evacuated.

2. Owing to the fact that measurements are taken at low pressures there is less possibility of errors due to deviations from Boyle's Law, the latter being most prevalent at high pressures.

3. Solubility errors are low owing to the small amount of reagent used and can even be completely eliminated if each reagent is evacuated before and after use. This may be accomplished by attaching a long piece of rubber tubing to the absorption pipette and lowering the reservoir thus effecting the release of all gases in the reagents.

4. Burette calibration errors are eliminated as volume measurements are not taken.

5. Errors due to supersaturated reagents are also eliminated as reagents are fresh each time.

6. A direct determination for nitrogen may be made when an iron oxide-copper oxide tube is fitted. This determination provides a useful check on the rest of the analysis in which the nitrogen is obtained by difference.

Description of the Apparatus

An isometric projection of the model is shown in the accompanying sketch. Mercury is used throughout as a gas-sealing medium and the base of the apparatus is so shaped to catch surplus mercury which may be recovered by opening the small drawer provided.

Similar to the Bone and Wheeler apparatus the gas burette consists of two similar parallel tubes, lettered A and B and enclosed in a water jacket. The tap situated at H controls the rate at which gas can be passed to the

particular reagents. The main reservoir may be raised or lowered either by hand or by the suction created by water pressure; reagents may be poured into the cup C and their volumes measured by the graduations at the top of the absorption pipette.

As a protection against accidental influx of mercury impairing the copper oxide a glass plate is fitted at O. The passage of gas to and fro over the copper oxide during oxidation is effected by means of a pump designed by V. E. Clifton.

Combustion tubes are heated either by a small electrical-resistance furnace or a gas furnace fitted with a governor and fine control valves.

Preparation of Reagents

1. POTASSIUM HYDROXIDE FOR CARBON DIOXIDE ABSORPTION. 25% BY WEIGHT.

2. ACTIVATED SULPHURIC ACID FOR UNSATURATED HYDROCARBONS

Concentrated H_2SO_4 with 1% of vanadium pentoxide or 0.6% silver sulphate. It is necessary to heat the acid fairly strongly to dissolve the vanadium pentoxide, but silver sulphate dissolves fairly easily. The above reagent is not the only one which may be used for determination of hydrocarbons; bromine water and fuming sulphuric acid will also do, but the chief objection lies in their attack upon mercury and consequent fouling of the gas burettes and passages.

3. CHROMOUS CHLORIDE FOR OXYGEN ABSORPTION

A permanently liquid amalgam is first prepared by mixing a few pieces of zinc and mercury in a glass-stoppered bottle. In the following order are added: 100 grams of chromous chloride, 260 ccs. distilled water and 40 ccs. concentrated HCl . The bottle is then shaken for from 5–10 minutes when the solution should become blue. Before the

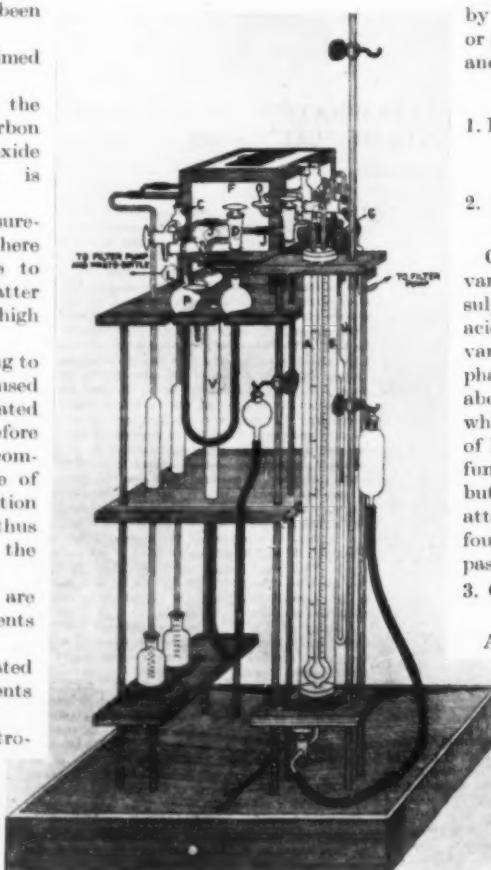


Fig. 1.—The G.L.C. Constant Volume Apparatus.

solution has a chance to oxidise it should be transferred to the absorption burette. A colour change from blue to green indicates that the reagent is fully oxidised and therefore useless from the point of view of further absorption of oxygen. It can, however, be regenerated by shaking again with zinc amalgam out of contact with air. One of the main advantages of chromous chloride over pyrogallol lies in the fact that the former does not evolve carbon monoxide during the absorption of large percentages of oxygen. The chromous chloride can be stored for a long time providing zinc and acid are added from time to time to replace loss due to reaction.

4. AMMONIACAL CUPROUS CHLORIDE FOR CARBON MONOXIDE ABSORPTION

Freshly prepared 11.5 grams cuprous chloride, 4.0 grams ammonium chloride, 40 ccs. 0.88 ammonia, and 50 ccs. distilled water.

Carbon monoxide may be estimated by absorption in the above solution and subsequent combustion of the remaining combustible gases, or it may be oxidised together with the hydrogen by passing over copper oxide (made from wire) at a temperature of 270°C. By means of the Clifton pump, the gas is passed to and fro over the oxide for a period of 15 mins.

No other aqueous reagents are required for the apparatus as ethane and methane are determined by passing over a catalyst of copper oxide-iron oxide at a temperature of 600°C.

The catalyst is prepared as follows: A mixture of ground copper oxide (99 parts) and ferric oxide (1 part) are added to 20 parts kaolin and water to form a paste. This is squeezed through a suitably sized tube to form threads which are broken into small lengths and fired at a temperature not exceeding 600°C. After packing well into the reaction vessel the latter is ready for operation.

From time to time both tubes of copper oxide-iron oxide and the low-temperature copper oxide should be regenerated by admitting air or oxygen.

Method of Operation

Vapour pressures are kept constant throughout an analysis by the use of a trace of dilute sulphuric acid in the gas tubes above the mercury levels. In a similar manner to that of the Haldane apparatus, air is blown through the water jacket to enable a constant temperature to be maintained in the gas burette. A gas sample stored

in one of the containers described in an earlier article may now be admitted by connecting to the tap at the top of the storage vessel. Traces of air are purged out of the space between the sampling tube and the gas burette by lowering the reservoir, drawing gas in, and ejecting through the absorption pipette. After this operation has been repeated a few times the gas is drawn into the burette together with mercury from the absorption pipette. A large volume of gas is chosen and the mercury adjusted to about half an inch from the burette tap.

The level is also adjusted to the zero in column A to enable a pressure difference reading to be made. Throughout an analysis the mercury in column A is repeatedly brought to the same arbitrary mark as initially made and the water jacket temperature read: in this way, the gas is kept at constant volume and only pressure changes are measured.

Potassium hydroxide is poured into the cup C, shaken in the absorption pipette and rejected with the exception of 1.0 cc. through the filter pump. By raising the main reservoir, gas is then passed together with a little mercury into the absorption vessel and the tap D closed. After shaking for about 3 mins., the gas is drawn back into the burette, the tap D again closed and pipette and capillary gently tapped to release air bubbles. After adjustment the KOH is rejected by means of the pump and waste bottle, and the cup, pipette, and capillary are washed with water followed by 10% sulphuric acid. Mercury is now pulled into the burette and adjusted to the mark at the top and the 0 mm. at the bottom. Pressure and temperature readings are taken and recorded.

Similarly, unsaturated hydrocarbons and then oxygen may be removed by shaking the gas in contact with, firstly, activated sulphuric acid,

SPECIMEN RESULTS OBTAINED FROM A COKE-OVEN GAS ANALYSIS

	Temperature °C.	Pressure mm.	Corrected pressure mm.	Contraction	Gas	%
Original	20.7	562.9	562.9	—	—	—
After KOH	20.7	548.4	548.4	14.5	CO ₂	2.6
After H ₂ SO ₄ + AgSO ₄ ...	20.7	532.3	532.3	16.1	Cn Hm	2.9
After CrCl ₃	20.7	529.7	529.7	2.6	O ₂	0.5
After CuO at 270°C. ...	20.7	202.6	202.6	327.1	H ₂	58.1
Plus N ₂	20.8	321.7	321.8	49.6	CO	8.8
After KOH	20.8	272.1	272.2	10.1	C ₂ H ₆	1.8
After CuO-Fe ₂ O ₃ at 600°C.	20.8	282.2	282.3	118.0	CH ₄	20.9
Plus N ₂	20.8	372.8	372.9	24.9	N ₂	4.4
After KOH	20.8	234.6	234.7	—	—	—

CH₄ = 372.9 - 234.7 - 2 (282.3 - 272.2) = 118.0 mm.

N₂ = 234.7 - (372.9 - 282.3) - (321.8 - 202.6) = 24.9 mm.

TEMPERATURE CORRECTIONS

Add to the pressure reading the following mm. corrections for a rise in temperature degrees C.

Burette differences mm.	0.1°	0.2°	0.3°	0.4°	0.5°	0.6°	0.7°	0.8°	0.9°	1.0°	2.0°
700	0.2	0.5	0.7	1.0	1.2	1.4	1.8	1.9	2.2	2.4	4.8
650	0.2	0.4	0.7	0.9	1.1	1.3	1.7	1.8	2.0	2.2	4.5
600	0.2	0.4	0.6	0.8	1.0	1.2	1.5	1.7	1.9	2.1	4.2
550	0.2	0.4	0.6	0.8	1.0	1.1	1.3	1.5	1.7	1.9	3.8
500	0.2	0.4	0.5	0.7	0.9	1.0	1.2	1.4	1.5	1.7	3.5
450	0.2	0.3	0.5	0.6	0.8	0.9	1.1	1.3	1.4	1.6	3.1
400	0.1	0.3	0.4	0.6	0.7	0.8	0.9	1.1	1.3	1.4	2.8
350	0.1	0.2	0.4	0.5	0.6	0.7	0.8	1.0	1.1	1.2	2.4
300	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.0	2.1
250	0.1	0.2	0.3	0.3	0.4	0.5	0.6	0.7	0.8	0.9	1.7
200	0.1	0.1	0.2	0.3	0.4	0.4	0.5	0.6	0.6	0.7	1.4
150	0.1	0.1	0.2	0.2	0.3	0.3	0.4	0.4	0.5	0.5	1.0
100	—	0.1	0.1	0.1	0.2	0.2	0.2	0.3	0.3	0.3	0.7
50	—	—	0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.2	0.3

and secondly, chromous chloride. The gas now ready for the hydrogen and carbon-monoxide determination is passed to the storage tube V. The copper-oxide tube is evacuated by using the mercury as a pump and nitrogen released through the absorption pipette. The Clifton pump is now brought into operation and the gas passed to and fro over the copper oxide at a temperature of 270° C., after which the copper-oxide tube is evacuated and the pressure of the gas measured after bringing to the constant volume mark.

Some nitrogen is now added to sweep the copper-oxide tube. Carbon dioxide formed during the reaction is removed by absorption in KOH, after which the gas is again brought to constant volume and pressure and temperature readings taken.

It may be assumed for all practical purposes that the only remaining combustible gases consist almost

entirely of methane and ethane. These are removed by passing to and fro over the copper oxide-iron oxide tube at a temperature of 600° C. for about 7-10 mins. Readings of pressure and temperature are again taken and nitrogen is added to sweep out the reaction tube. Carbon dioxide is again removed and readings taken as before. An increase in pressure after combustion is indicative of ethane present as the latter gas produces twice its volume of CO₂ whereas pure methane would produce only its own volume.

Water jacket temperatures are recorded with pressures, and in the event of a change in temperature, a correction is made to the particular pressure by consulting a table supplied with the instrument.

If the results are to be expressed as total paraffins, C_xH_{2x+2}, the increase in pressure after combustion is subtracted from the total CO₂ produced

and the value of "x" determined by dividing the CO₂ by the paraffins.

A specimen sample of results obtained during an actual analysis of a coke-oven gas illustrate the method of determining the percentages of the constituents. Although developed primarily for the determination of gaseous mixtures formed during carbonisation processes, the apparatus can be used for all manner of gases—e.g., exhaust gases from boiler flues, furnaces, etc., natural gas, acetylene, blast-furnace gas, producer gas, hydrogen, etc. There seems to be no limit to its adaptability and where accuracy is essential the instrument has much to recommend it.

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A Split-Type Micro-Combustion Furnace

By G. Ingram

A split-type combustion furnace is described which can be constructed cheaply from materials readily available.

ALTHOUGH designs of split-type furnaces have been published in the American literature^{1, 2} over a number of years, none of British design is available in this country. Their high cost of manufacture, coupled with the fact that the demand is not too great, despite advantages over other forms of heating, may well account for this deficiency. An attempt has been made by the present author to produce a satisfactory furnace of the split type which can be manufactured cheaply with material readily available to most microchemists in this country.

The main drawback of electrical forms of heating is essentially the high cost involved, in order to produce the requisite refractory of the desired shape which also incorporates the housing for the element wire. This involves the casting of moulds and the subsequent firing of the refractory on lines that do not allow any individuality in the design of the ultimate furnace. The preparation of a shaped refractory such as that required for a split-type furnace can therefore under these conditions only be produced with some difficulty. Recent developments in the preparation of refractory materials have eased the position greatly. Insulating brick can now be obtained, having properties which allow shaped refractory blocks to be produced cheaply. The material used in the micro-combustion furnace described below, in the form of a brick, can be sawn,

filed, machine-tooled and drilled without any appreciable fracturing. These properties simplify the design of the furnace and allow fixing of the element wire so that failures due to burnt-out elements can be remedied quickly.

The furnace which is suitable for Dumas, halogen and sulphur determinations, consists of four coils of oxidised Nichrome V. wire housed in alumina refractory tubes which are encased in the shaped refractory block. The latter is shaped to allow the side insertion of the combustion tube and is enclosed in a welded metal case. The alumina element tubes fit into the block so that they can be withdrawn from one end to facilitate replacement of the element coils.

Materials Required

1. 0.048-in. steel sheet for the case.
2. Gibbons (Dudley) "HT1" refractory insulating brick.
3. Alumina refractory tubes, 9 mm. outside dia. (Cat. No., Morgan Crucible Co. "Triangle H." T7681).
4. Element wire. Nichrome V. oxidised wire, 0.0179 in. dia., 6.31 ohms per yd.
5. Material for control box (Metal or "Syndano").
6. Sunvic "Simmerstat" type "TYC," and 0-5 amp. range ammeter.

Construction of the Furnace

The metal case is constructed from .048 in. steel sheet and welded to form an oblong box (Fig. 1A),

1. Hallet, L. T. *Ind. Eng. Chem., Anal. Ed.*, 1938, 10, 101.

2. Other types of split furnaces are described in "Standard Methods of Chemical Analysis." Scott and Fifer, Vol. II, 5th Ed., 1939.

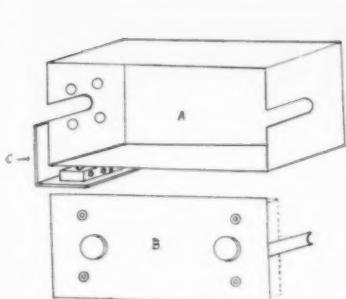


Fig. 1.—Furnace case.

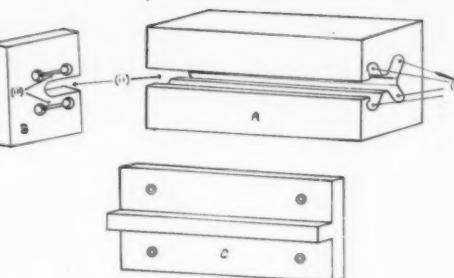


Fig. 2.—Furnace refractory block.

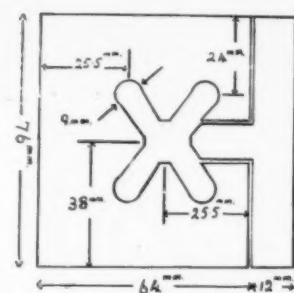


Fig. 3.—Cross section of refractory block.

177 mm. long, 76 mm. wide and 76 mm. deep. Slots, 12 mm. wide are cut from the open edges to the centre of the two ends, and four 9 mm. dia. holes are drilled at the back end of the furnace case. Another piece of steel sheet 177 mm. long and 76 mm. wide is cut as shown in Fig. 1B to form the movable lid of the furnace. Two porcelain connection blocks are fixed with 4 B.A. hank bushes and screws to the underside of the case as shown and are protected with a metal plate (Fig. 1C) fixed to the back end of the metal case with two hank bushes and screws.

The support for the furnace is adjustable. It consists of a 6 mm. thick brass plate 5 cm. in dia. which has a supporting rod 14 mm. in dia. and 5 cm. long welded to it. This support is screwed to the underside of the metal case with three 4 B.A. hank bushes and screws. The short protruding rod fits into an adjustable clamping tube fixed to the top of the control box. Both this and the rod have a hollowed centre to allow the passage of the mains leads from the furnace to the control box.

The refractory block is shaped to fit the assembled metal case and is composed of three parts: (a) (Fig. 2), the main portion is approximately 160 mm. long, 64 mm. wide and 76 mm. high; (b) an end plate, 14 mm. long, 64 mm. wide and 76 mm. high; (c) the section attached to the metal lid which is shaped to fit snugly into the open front of the furnace.

The main section (Fig. 2A) is shaped in the following manner. Four 9 mm. dia. holes (i) which are required to house the alumina element tubes are drilled through the entire length of the block to correspond with the holes in the back end of the metal case. The groove (ii) 12 mm. wide is then cut into the front face of the block to a depth of 32 mm., and connecting channels to the four drilled holes produced by careful cutting out, using a thin flat file. The shape shown by the cross-sectional drawing of the block (Fig. 3) is then obtained by further filing with a flat file. One of the alumina tubes is then inserted in turn into each of the channels (i) and moved back and forth until a loose fit is obtained.

The end plate (B) is situated at the front end of the furnace in order to provide an insulating block for the connecting wires of the element coils and as a means of ensuring a rigid support for the ends of the coil tubes. The plate is slotted to correspond with the main section and four holes are drilled to a depth of 8 mm. to coincide with the alumina tube channels. Each pair of holes above and below the slot is connected by a narrow groove (iii) in which the coil-connecting wires seat when they are inserted into the furnace.

The refractory lid has the shape shown (Fig. 2C) and is fixed to the metal lid with four 4 B.A. hank bushes and

screws. Because the lid of the furnace is fairly tight fitting no means of fixing is needed. To facilitate its removal, small heat-resisting handles are fixed at both ends of the lid.

The finished sections of the refractory block are fixed within the metal case with alumina cement.

The length of each of the 7 mm. bore alumina tubes is 177 mm. These lengths are cut off from the supplied lengths of the tubing by simply scratching a line with a powered grindstone and snapping off the required piece. The tubes project from the back end of the furnace about 6 mm. to facilitate their removal in order to insert the element coils.

Elements for the furnace are prepared by winding sufficient wire on to a $\frac{3}{16}$ -in. metal rod fixed in a lathe to form a coiled length having an approximate resistance of 120 ohms. The coil is cut into two equal parts. Each resulting coil is then divided at its central point and three of its component coils pulled out to form two coils, connected by a short length of straight wire. Connecting wires are obtained by pulling out a few of the coils from each of the ends of the elements. After the elements have been placed inside the furnace, the main connecting wires are covered with insulating beads and then connected up to the porcelain blocks situated beneath the furnace case.

The elements are inserted into the furnace in the following manner. With the alumina tubes withdrawn, one set of coils is inserted into the two lower channels (i) so that their short length of connecting wire drops into the narrow groove (iii) (Fig. 2B) and the two straight wires pulled out from the free ends protrude through the two lower holes in the end of the metal case. The alumina tubes for the two lower coils are then pushed over the coils so that their ends seat firmly in their respective holes bored in the refractory end plate. The other set of coils is then inserted into the top channels in a similar manner and covered with their alumina tubes.

The separate coils are connected in series to the main leads which proceed through the hollowed centre of the furnace support, to the "Simmerstat" control situated in the box below the furnace.

The controls are enclosed in a box constructed mainly with "Syndanyo" heat-resisting material. The box is 177 mm. long, 100 mm. wide and 100 mm. high. The top, bottom and the two ends are cut out from 12 mm. thick board, and the back and front from aluminium sheeting. The various parts are fixed together with screws and the front metal panel is drilled to hold the ammeter, Sunvic relay and a 5 amp. mains switch.

New Equipment

Protective Equipment for Handling Radium Sources in Industrial Radiography

AMONG the various non-destructive methods developed for the testing and inspection of castings, welded assemblies, soldered or brazed joints and many engineering components, that employing gamma-rays is now firmly established in industry. For this purpose suitable radium and radon sources are now obtainable from the Government-owned Radiochemical Centre at Amersham. For many years, Johnson, Matthey and Co. Limited, have maintained a laboratory specially equipped and staffed to carry out research in this field and to develop improved radiography techniques. Particular attention has been given to the question of safety and to devising methods of reducing to the very minimum the amount of radiation to which the operator is exposed. As a result of the experience gained in this work, a number of special items of equipment have been designed which have proved efficient and, at the same time, simple to operate. Arrangements have now been made to manufacture these in quantity so that they may be generally available. Instructions may thus be given to the Radiochemical Centre for sources to be sent to the Johnson Matthey laboratories at Wembley for fitting into approved carriers. In this way the user avoids the major risks associated with the handling of unscreened radio-active materials.

The Portable Carrier

The bulk of industrial radiography is carried out by means of 100-250 mgm. radium sources, whose use demands every possible precaution. Satisfactory results may sometimes be obtained from sources as small as 15-30 mgm., but these can obviously handle only a limited range of inspection work.

To house the larger and more commonly used sources of 100-250 mgm. a standard portable carrier (Fig. 1) has been devised, suitable for both the storage and transport of the radium. It weighs only 38 pounds and



Fig. 1.—This portable carrier is designed to serve both as a convenient source for radiography and as a storage unit offering satisfactory protection.

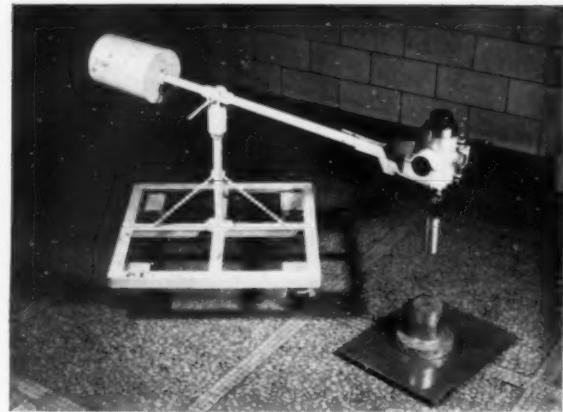


Fig. 2.—The mobile stand enables the easy handling of the source in its protective carrier. The sighting device, used before exposure begins, ensures correct alignment on to the object.

can thus be handled with ease by one man. The protection given by this equipment is extremely good and at a distance of 3 ft. from a carrier containing a 250 mgm. source, the radiation is less than 0.2 röntgen per day, well below the accepted tolerance dose. This carrier can also be used to house radon or other gamma-ray sources up to a value of 250 millieuries.

The carrier is machined from a phosphor bronze casting and incorporates a thick liner of copper-tungsten Elkonite, which has a very high coefficient of absorption of radiation. The radium source, in a platinum container, is held at the centre of the carrier by a clip mounted upon an Elkonite rod. Extraction of the radium is easily achieved by means of a handling extension which, after the knurled cap at the top of the carrier has been removed, can be screwed into the rod bearing the source. Two heavy conical plugs are fitted in the ends of the carrier, and these are so designed that the operator's hand is not exposed to direct radiation while they are being removed or replaced.

Mobile Stand and Sighting Device

In making single exposures the portable carrier may be mounted upon a mobile stand, shown in Fig. 2, for which an accessory device for accurate sighting is manufactured. The design of the carrier is such that upon the removal of one of the conical plugs a unidirectional radiation beam of fairly narrow angle is released. After the sighting device has been used to bring radium source, carrier orifice and object exactly into line, the conical plug is removed and the exposure made.

Time-Controlled Exposure Unit

Panoramic exposures, by means of which the radiography of 40 or more castings may be carried out simultaneously, are made by combining the portable carrier with a time-controlled exposure unit (Fig. 3). This, essentially a small motor driven lifting device, may be set to operate at a certain time up to seven days in advance, and to make an exposure of predetermined duration. The operating rod of the unit is screwed into the rod which supports the source, and at the chosen time lifts the radium from the carrier to a height of roughly 7½ in. from the floor. When exposure is complete

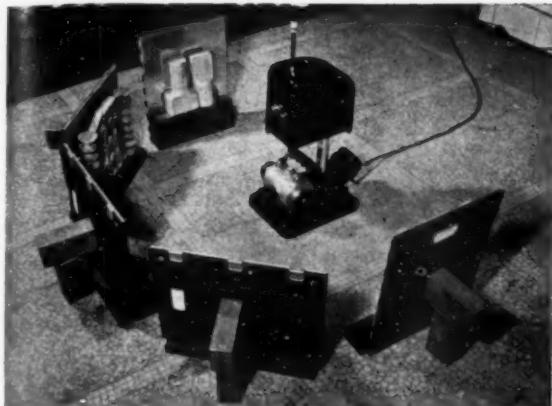


Fig. 3.—The time-controlled exposure unit is designed to work in conjunction with the standard carrier and is employed in panoramic exposures when a number of objects may be radiographed at one time.

the source is automatically returned. By use of this unit, it is simple to ensure that operators are at no time subjected to harmful radiation. Exposures can be timed to take place in a cleared room at any convenient time of the day, or during the night. A button switch for manual operation of the unit is fitted, and in addition there is a warning light which operates only when the source is exposed. Sockets are provided to allow the extension of the light circuit, and a remote warning system may thus be easily arranged.

White Magnetic Fluid

A New Development in Magnetic Crack Detection

MAGNETIC crack detection—in which a "magnetic fluid or detecting ink" is applied to magnetised components by immersion, pouring or spraying methods—is now firmly established as a means of finding surface cracks, flaws or fissures. The magnetic fluid is a suspension of magnetic particles in a base such as paraffin or methylated spirits, the magnetic particles being attracted to the edges of a crack.

Important features of the fluid are good colour contrast between the particle formation and the surface of the article under test, and slow gravitation of the par-

ticles in the suspending medium. Magnetic fluid containing black particles is, therefore, most successful on brightly finished surfaces, but is unsuitable for use on dark surfaces such as those found on forgings, stampings, "black" bar, etc., unless colour contrast is obtained by the application of white paint before testing. Alternatively, a fluid containing coloured particles, for example, the red magnetic iron oxide, which form a contrast with the dark surface may be used. However, where a black and white contrast is considered best for dark surfaces, then a white magnetic fluid is very desirable and, in fact, the demand for such a fluid has persisted in spite of the existence of the red fluid.

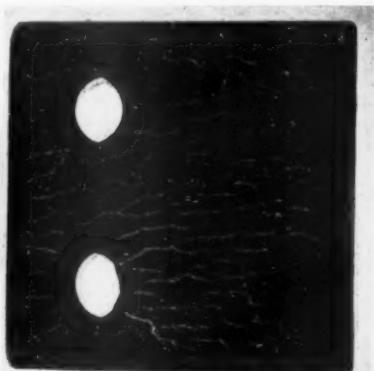
In the past experiments with nickel powder in paraffin have been made in attempts to produce white magnetic fluid. Although these were successful magnetically, the grey colour and rapid gravitation due to the greater density and particle size rendered the method impracticable. Mixtures of a white powder, such as French chalk, and normal black magnetic fluid failed since most white powders darken on wetting with paraffin.

The new fluid developed in the Research Department of the Metropolitan-Vickers Electrical Co. Ltd., employs a white additive or "tracer" which retains its whiteness when immersed in paraffin. Whilst any powdered light-coloured metal of low density would suffice, the use of finely divided aluminium has been remarkably successful.

Tests with specimens known to contain cracks have shown that there is no reduction in the sensitivity of the fluid due to the addition of the aluminium powder. Surface cracks are shown as white lines, due to the presence of the aluminium powder carried to the defects along with the magnetic particles; the contrast is greatly improved by allowing time for draining.

The accompanying illustrations show the appearance of hardening and grinding cracks as revealed by the new fluid. In both cases the residual magnetism was sufficient for crack detection. In another test, longitudinal cracks in a hexagonal "black" bar were detected with the white fluid, using a pouring action, even when the cracked region was on the underside. Sub-surface defects may be found, and even on brightly finished surfaces appreciable colour contrast is obtained.

Where large numbers of components such as unmachined forgings are being crack-detected by the white-paint black-fluid technique, the substitution of the white magnetic fluid method should save valuable time and ultimately reduce the cost of inspection.



Left—quenching crack in small shaft. Right—typical grinding cracks. Both these parts have been crack detected by the magnetic method using the white magnetic fluid described above.

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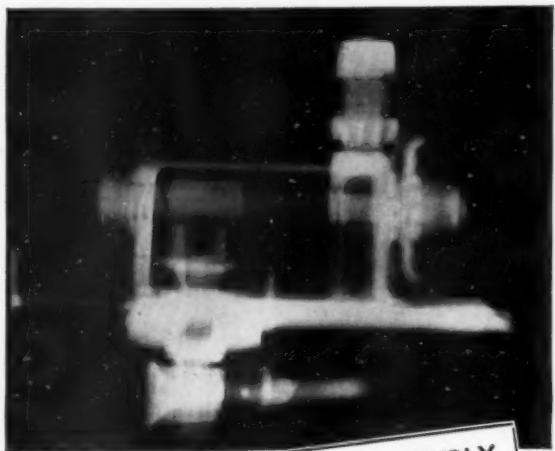
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Electronic Instruments Symposium and Exhibition

THE first Electronics Symposium of the Scientific Instrument Manufacturers' Association, held a year ago, was such an outstanding success, and evoked so many expressions of appreciation of the useful purpose served by it, that the Association had no hesitation in repeating the venture this year. The second Symposium on Electronic Instruments in Research and Industry, together with an Exhibition of equipment by members of the Electronics Group of the Association, was held at the Examination Hall, Queen Square, London, W.C.1, on November 2nd, 3rd and 4th.

Technical Papers

Following the official opening by Sir George Thompson, F.R.S., on Wednesday afternoon, there were four sessions for the presentation and discussion of papers covering various aspects of electronic instrumentation. These were of a varied nature and comprised the following :—

“ Electronic Instrumentation in Atomic Research,” by Denis Taylor, M.Sc., Ph.D., M.I.E.E., F.Inst.P. of A.E.R.E., Harwell.

“ Electronic Amplifiers,” by I. L. Banks, S.S.M.C., of Furzehill Laboratories, Ltd.

“ Magnetic Amplifiers,” by A. V. Hemingway, B.Sc. (Eng.), A.M.I.E.E., of Elliott Bros. (London), Ltd.

“ Some Recent Improvements in Electronic Measuring Techniques,” by C. H. W. Brookes-Smith and J. A. Colls, B.Sc., A.R.C.S., of Southern Instruments, Ltd.

“ The Measurement of Some Transient Phenomena,” by H. A. Dell, Ph.D., of Mullard Electronic Instruments, Ltd.

“ An Industrial Servo Mechanism,” by P. H. Briggs, B.Sc. (Tech.), of Ferranti, Ltd.

“ Co-operative Research at B.S.I.R.A.,” by A. J. Maddock, D.Sc., F.Inst.P., M.I.E.E., of the British Scientific Instruments Research Association.

The Exhibition

In view of the restricted number of papers which can be presented at such a Symposium, it is obvious that only a limited number of industries can be covered each year. In planning the accompanying exhibition, therefore, the instruments to be displayed were chosen so as to be of interest to as wide a field as possible. Whilst many of the exhibits of general interest, such as resistance, current and voltage measuring instruments; oscilloscopes; amplifiers; cameras; and galvanometers; find application in the metallurgical laboratory, limitations of space prevent anything more than a passing reference being made to all but a few items of particular interest in the metallurgical field.

Analytical Equipment

On the analytical side, BAIRD AND TATLOCK (London), Ltd., included in their exhibits a B.T.L.-Mullard potentiometric titration apparatus, a photo-electric turbidimeter, electrolytic analysis apparatus and a number of balances. The potentiometric titration apparatus consists of a titration unit—a heavy cast-iron stand, finished in stoving enamel and fitted with two

burette holders, electrode holder, stirrer and motor, hot-plate and “ Magic Eye ” balance indicator—and a potentiometer unit. A “ Magic Eye ” electron beam tuning indicator is also included in this unit, thus obviating the use of a delicate galvanometer. There are no batteries and a special circuit eliminates all chance of drift during titration. Supply voltage variations do not give rise to any inaccuracies. The turbidimeter was originally designed for the measurement of the turbidity of water supplies but there is no reason why it should not be used for other purposes, including analytical work. It is claimed that the apparatus is suitable for the rapid determination of sulphur by measuring the turbidity of the suspension of cadmium sulphide, formed by passing the hydrogen sulphide evolved from the steel into cadmium acetate solution, as in the usual volumetric sulphur method. For those determinations which involved the electro-deposition of the element to be determined a compact set-up which includes a built-in hot-plate was exhibited.

The Spekker photo-electric Absorptiometer, made by ADAM HILGER, LTD., has now firmly established itself in those fields of chemical analysis where colorimetry is employed. On view at the exhibition was the latest model, the result of a complete re-design. The scale length has been increased and a moulded plastic cell-carrier and a sliding filter carrier are now incorporated.

MULLARD ELECTRONIC PRODUCTS, LTD., included a Conductivity Bridge in their exhibits. With three ranges, 0.1-10, 10-1,000, and 1,000-100,000 micro-mhos, the instrument employs a bridge circuit with “ Magic Eye ” balance indicator.

For those interested in pH measurement, MARCONI INSTRUMENTS, LTD., had on show a pH meter.

Included in the display of electronic instruments on the Stand of AIRMEC LABORATORIES, LTD., was a model of one of their most recent developments, a material comparator which can be used for sorting mixed materials of the same size or as a dimensional check on a particular material.

Temperature and Strain Measurement

On the Stand of SUNVIC CONTROLS, LTD., various types of vacuum switches, energy regulators, time-delay relays, and bimetal thermostats were displayed. For creep-test furnaces and other furnaces where accurate temperature control is required the Sunvic electronic, proportioning resistance-thermometer controller was displayed. By means of this instrument, used in conjunction with a nickel or platinum resistance-thermometer, the average power applied to the furnace is proportional to the extent to which the bridge is out of balance. At full sensitivity the circuit will operate on a charge of resistance of one-part in 5,000 and at the same time give proportionate control without moving parts. In thermocouple and strain-gauge work, the accurate measurement of small D.C. e.m. fs. is a day-to-day problem. The Sunvic D.C. Amplifier has been developed with a view to meeting the needs of workers in these fields. The stability and speed of the amplifier makes it convenient to use in conjunction with auxiliary

switchgear to measure and record the output of a number of thermocouples or strain-gauges, as opposed to the more tedious method of measurement by potentiometer. For the measurement of furnace temperature in creep-test work the instrument enables a full scale record to be obtained for a temperature change which is extremely small in relation to the operating temperature.

A four-channel-strain recorder on which a permanent record of four different strain gauges can be obtained was shown by KELVIN AND HUGHES (Marine) LTD.

Supersonic Flaw Detection

The use of the supersonic method of detecting internal flaws in metals has grown rapidly in recent years. The method depends on the fact that a flaw in the path of a beam of supersonic energy reflects at least a part of the energy, the reflection being "received" and indicated on a cathode ray oscilloscope. A recent development shown by KELVIN AND HUGHES (Industrial), LTD., was a set of 'transverse wave' probes for butt-weld testing. The transmitter probe directs a supersonic wave beam, in the form of pulses of short duration, at a shallow angle towards the weld seam. The beam approaches the weld after reflection from the other side of the parent metal and any flaws are indicated by reflections picked up by the receiver probe, which is located on the same side of the weld as the transmitter. It is important to use a single beam of supersonic waves and for this purpose a perspex probe generating transverse waves has been devised. These waves propagate at half the velocity of the longitudinal waves normally employed, the wavelength also being halved. This leads to a beam of comparatively high sensitivity and directionality. Any thickness of plate can be tested, although below $\frac{1}{2}$ in. detailed analysis of flaws becomes more difficult. For general supersonic flaw detection a high power transmitter unit is now available for use in cases where the energy available from the standard set is insufficient, due to large grain size, rough surfaces or excessive length of path.

Gauging in Sheet Rolling

For measuring the reduction in thickness given to steel strip in "the temper pass" the BALDWIN INSTRUMENT Co., LTD. showed an instrument based on magnetic principles. An A.C. generator is driven from the rolls and its output fed to two "printing" coils which are displaced relatively to each other across the width of the strip. That on the ingoing side is adjustable by a micrometer and the other one fixed. The coils produce a magnetic "pattern" on the steel, the form of which is dependent on the roll speed. Two "pick-up" coils, are in line with each printing coil, pick up the patterns which after amplifying, are fed to a phase selective indicating detector. The ingoing "printing" coil is set to the required distance so that, for the correct reduction, the phase detector indicates zero. Any deviation from the correct reduction will then be indicated as a deflection of the indicator.

TAYLOR, TAYLOR AND HOBSON, LTD., exhibited their latest model Continuous Gauge, Type 'E' ("Flying Mike") in which the metal strip passes between two rollers set to the correct gauge thickness. Any variations are shown on a micro-ammeter scale and adjustments to the mill can be made accordingly. While the gauge is normally used as an indicator, records may be obtained on a 'Talymin' recorder.

Also displayed by this firm was the latest Model 3

Talysurf surface measuring instrument which is an improvement on previous models. The results are obtainable in the form of graphs or as readings on a simple average meter. A unique new feature is the pen recorder which produces a graph having rectilinear co-ordinates. At the same time the use of ink has been superseded by electrical marking on Teledeltos paper. The new model is also suitable for taking measurements on roll surfaces, etc., without removal from the machine.

Vacuum Equipment

W. EDWARDS AND CO. (LONDON), LTD., have long specialized in high vacuum equipment and included in their display were rotary and oil and mercury diffusion pumps, small compressors, Philips and Pirani gauges, ionization gauges and various forms of valves for use in vacuum work. Of particular interest to research metallurgists was a small coating plant which can be used for, among other purposes, shadow casting on electron microscope replicas in order to determine more accurately the topography of the etched surface.

Preparing Thin Specimens for Creep and Tensile Testing

By PAUL FELTHAM, B.Sc., A.Inst.P.*

IT is often desirable and, in view of the lack of heavy equipment, sometimes necessary to carry out creep or tensile tests on a small scale in the laboratory.

If wires or rods less than about 0.1 in. in diameter are to be used for making the specimens, great difficulties are generally experienced in machining them into a suitable shape with enlarged ends.

The following method has been used successfully in producing mild steel specimens about 0.05 in. in diameter suitable for creep experiments under constant stress in the temperature range 950°-1,400° C.

The ends of a length of rod of the metal about 0.065 in. in diameter were covered with sealing wax so as to leave just a little less than the gauge-length free. The rod was then kept in motion in a solution of about 10N sulphuric acid until the exposed part had diminished to within about 0.002 in. of the required diameter. The etchant was found to have attacked the metal uniformly so that, after the removal of the wax, it was only necessary to finish the ends of the rod (where the uncovered and the previously waxed portions meet) on a watchmaker's lathe, and to polish the entire specimen in the usual way before it could be inserted into pin-vices and used.

* British Iron and Steel Research Association.

The Institution of Metallurgists Examination, Pass List, 1949

Licentiateship: Cockle, J. E. S. (Beckenham), Harwood, E. S. (Birmingham), Moore, J. P. (London), Savage, T. R. (Sunderland), Ward, E. (Doncaster), Watson-Adams, B. R. (Ashford), Miodownik, A. P. (London).*

Associateship: —Allon, F. R. H. (Southampton), Dunthorne, H. B. (Slough), Hull, W. G. (Slough), Morton, G. R. (Kettering), Wilson, A. (Glasgow).*

Fellowship: Evans, F. C. (Gerrards Cross).

* Election awaits compliance with age and experience regulations.

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